THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

DECEMBER, 1958

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Determination of the Die-Filling Characteristics of Metallic Powders

J. OAKLEY: 'A Method of Assessing the Die-Filling Characteristics of Powders.'

Jnl. Inst. Metals, 1958, vol. 4, Sept., pp. 26–8.

Standard methods of determining the flow characteristics and apparent density of metal powders have serious limitations when used to obtain data of value in tool design. With the introduction of new presses and the demand for parts of more complicated design, the need has arisen for a means of determining the filling characteristics of powder mixtures which takes into account the effect of the physical dimensions of the die space to be filled. This paper gives details of a test unit designed to meet this need.

The apparatus consists of a powder container which can be traversed over a recessed hole, in which are a series of cups of different capacities. The cups, which may contain a core, are intended to simulate the cavity in a normal tooling set-up. Filling density is determined from the weight of the powder which has entered the cup after the powder container has been traversed over it. The results obtained are illustrated by data derived from tests with, *inter alia*, stainless-steel powders and various grades of nickel powder.

Applications of E.D.T.A. in Metallurgical Analysis

E. G. BROWN: 'Ethylenediamine Tetra-Acetic Acid and Allied Compounds in Metallurgical Analysis.'

Metallurgia, 1958, vol. 58, Sept., pp. 149–59.

The article reviews the considerable amount of literature recently published on the analytical applications of the 'Complexones' in the metallurgical field. The survey is supplementary to that published by the author in *Metallurgia*, 1954, vol. 49, Feb., pp. 101–5; March, pp. 151–5 (*Nickel Bulletin*, 1954, vol. 27, No. 5, p. 80).

The review, which is based on a *bibliography* of 83 items, and is believed to include substantially all of the available literature, is in two sections. The first covers analysis of non-ferrous materials, and gives the salient features of new or modified procedures for determination, in specific materials, of aluminium, beryllium, bismuth, cadmium, cerium, chromium, cobalt, copper, gallium, gold, indium, iron, lead, magnesium, magnesium-oxide, manganese, molybdenum, nickel (in Raney nickel and high-nickel alloys), selenium, silver, thorium, tin, titanium, uranium, zinc and zirconium. The second section refers to techniques suitable for determination, in steels, of aluminium, beryllium, cobalt, copper, magnesium, manganese, selenium and silver.

Lap Seam Welding Equipment and Technique

See abstract on p. 342.

NICKEL

Smelting and Refining of Nickel-containing Ores

Engineering and Mining Journal, 1958, vol. 159, Mid-June issue, is devoted to the 1958 edition of the *Mining Guidebook*, the fifth in a series of annual publications reviewing developments in prospecting, mining, milling, smelting and refining techniques.

A major part of the *Guidebook* is taken up by the section on mineral processing. The thirty-two flowsheets presented summarize procedures developed to overcome the problems peculiar to each of the wide range of mineral deposits considered. Each particular processing problem is stated, an annotated flowsheet represents the solution evolved, and finally details are given of the results obtained. References to flowsheets pertaining to nickel-containing ores are given below.

p. 71: Recovery of cobalt from nickel-refinery residual solutions. (Sherritt Gordon Mines, Ltd.)

p. 73: To refine nickel and cobalt from a sulphide concentrate, use is made of an ammonia leach to form soluble nickel, cobalt and copper sulphates, which are then selectively recovered. Nickel is reduced to metal by hydrogen treatment. High-purity nickel powder and briquettes are produced, and the recovery of nickel is higher than 90 per cent. (Sherritt Gordon Mines, Ltd.)

p. 74: Recovery of base metals from arsenical-nickel-sulphide flotation concentrate by dry chlorination. (Mitterberg Mining Co.)

p. 89: Flowsheet for treatment of high-arsenical-sulphide cobalt and silver concentrates and

ores, giving commercial recovery of silver, nickel oxide, cement copper, cobalt oxide, cobalt, refined arsenic and a Dore bullion. (Deloro Smelting and Refining Co., Ltd.)

p. 94: Two flowsheets (one of the smelting and refining processes used in 1918, the other of those in present use) emphasize the advances made by The International Nickel Company, Inc. in processing copper-nickel-cobalt ores and concentrates, and illustrate the complexity of the procedures which have been evolved to extract and refine the 14 metals produced by the Company.

p. 95: Flowsheet indicating the salient features of the electrorefining technique developed by The International Nickel Company, Inc. for production of nickel from nickel-sulphide anodes (see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 8, p. 213).

Electrolytic Separation of Nickel and Cobalt

M. J. FERRANTE, P. C. GOOD and P. M. FRUZENSKY: 'Electrolytic Separation Studies of Nickel and Cobalt from Nicaro-Plant Residues.'

U.S. Bur. Mines, Report of Investigations 5394, May 1958; 30 pp.

In an effort to increase efficiency of nickel production at the Nicaro (Cuba) plant, where lateritic ores are treated, the U.S. General Services Administration recently initiated research projects with various organizations. This report covers one phase of the investigation, which dealt specifically with development of an electrolytic process for recovery of 'specification-grade nickel' (cobalt 1 per cent. max.) from Nicaro pregnant liquor or from the basic carbonate precipitate currently in production. The products to be treated contained nickel and cobalt in the ratio of 64:1.

Two main lines of investigation were explored: (1) separation of cobalt from nickel solutions, using chemical or electrolytic methods, and (2) selective leaching of nickel from basic carbonate precipitate containing nickel and cobalt, followed by recovery of nickel by electrodeposition. Prior to commencement of experimental work an exhaustive search was made of the literature, involving consultation of some 250 items: details of immediately relevant references are given in the report.

Research at The Birmingham Laboratory of The Mond Nickel Company, Ltd.

See abstract on p. 330.

Thermal Fatigue of Nickel

H. MAJORS: 'Thermal and Mechanical Fatigue of Nickel and Titanium.'

Amer. Soc. Metals, Preprint 105, Oct. 1958; 12 pp.

It has been observed that in thermal cycling the behaviour of a brittle material differs from that characteristic of a ductile metal. In this report the author describes thermal and load-cycling tests made on 0·500-in. diameter thin-walled tubes of titanium and nickel, under conditions involving direct loading and constant mean temperature. The machines used in the tests were similar to those designed by other investigators, in order that the results might be strictly comparable: machines and test pieces are described and illustrated.

The titanium specimens were tested at a mean temperature of 575°F. (302°C.) and the nickel at 525°F. (274°C.), temperatures chosen to avoid any phase changes in the metals during the cycling either of load or of temperature. During thermal fatigue the frequency of cycling was 100 and 120 cycles per hour for titanium and nickel, respectively: under load-fatigue cycling the frequency was 60 cycles per hour for both materials. Average grain size of the titanium was A.S.T.M. No. 7 and that of the nickel was No. 5.

Chemisorption of Nitrogen on Nickel

P. W. SELWOOD: 'The Mechanism of Chemisorption: Nitrogen on Nickel.'

Jnl. Amer. Chemical Soc., 1958, vol. 80, Aug. 20, pp. 4198-201.

As an introduction to report of experimental work, the author discusses the theory of nitrogen adsorption on nickel at low temperatures. The major literature is considered.

In the present investigation the magnetic method was applied to study of this phenomenon. It is demonstrated that nitrogen on nickel shows no true chemisorption in the sense in which that term is generally used. The adsorption of nitrogen on a nickel-silica catalyst at moderately low temperatures (−78°, −50° and −20°C.) produces a small reversible loss of magnetism as measured by the low-frequency a.c. permeameter method. A similar effect was observed with argon and krypton. It is considered that these effects, which cannot be due to molecular chemisorption, may be the result of polarization of physically adsorbed molecules, or of dimensional changes caused by the adsorbed gases, or of both effects.

Solid Solubility of Phosphorus in Nickel

J. KOENEMAN and A. G. METCALFE: 'The Solid Solubility of Phosphorus in Nickel.'

Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers, 1958, vol. 212, Aug., pp. 571-2.

The eutectic arrest was found to be in the region of 875°C., confirming earlier determinations by KONSTANTINOW (*Zeitsch. anorg. Chemie*, 1908, vol. 60, p. 405). A plot of log. solubility against the reciprocal of temperature (based on metallographic examination of aged specimens of low-phosphorus alloys) shows values lying on a straight line from about 0·04 per cent. at 500°C., extrapolating to about 0·17 per cent. at the eutectic temperature of 875°C.

Behaviour of Nickel in Contact with Fluorine

See abstract on p. 347.

Nickel Powders for Sintered-Plate Storage Batteries

The following abstract is published of a paper recently presented before The Electrochemical Society:

C. F. COOK, W. F. NYE and F. W. LEONHARD: 'Morphological Characteristics of Nickel Powders for Use in Sintered-Plate Nickel-Cadmium Storage Batteries.'

Jnl. Electrochemical Soc., 1958, vol. 105, Aug., p. 154C.

'The relationship of the morphological characteristics of nickel powders to plaque porosity, abrasion-resistance, sintering, and impregnating characteristics is revealed by stereo-electron microscopy, using a new replica technique. The powders studied include those prepared by thermal decomposition, electrolysis, and chemical precipitation. Unique among the nickel powders studied is carbonyl nickel, which exhibits a three dimensional dendritic structure with well-developed crystal faces. It is this characteristic which appears to give the material unique sintering properties.'

Moulded Nickel Cathodes

C. P. HADLEY, W. J. RUDY and A. J. STOECKERT: 'A Study of the Moulded Nickel Cathode.'

Jnl. Electrochemical Soc., 1958, vol. 105, July, pp. 395–8.

Early work on moulded cathodes was reported by MACNAIR, LYNCH and HANNAY, who described the fabrication of the cathodes and some of their properties, and made comparison between the emission of moulded cathodes, oxide cathodes and 'L' cathodes (*Jnl. Applied Physics*, 1953, vol. 24, p. 1335). The present authors give reference also to subsequent literature and, as a background to the report of their experimental work, they discuss the essential features of moulded cathodes. The design of a typical cathode is illustrated, and the following main features are noted.

'The cathode sleeve is a nickel cylinder having an outer diameter of 0·125 in., a length of about 0·33 in., and a wall thickness of 0·004 in. The cathode pellet is composed of two layers. The lower layer, about 0·045 in. thick, contains nickel powder and an activator (ZrH_2) in the weight proportions of 99:1. The upper part of the pellet, which is 0·003 in.–0·005 in. thick, contains nickel powder (69 per cent. by weight), alkaline-earth carbonates (30 per cent.) and an activator (1 per cent.). The nickel powder is fired at 600°C. for 15 min. in a hydrogen atmosphere (−10°C. dew point). All powders are thoroughly dried before use.

'The cathode pellet is formed and pressed into the sleeve at a pressure of 80 tons/sq. in., in a single step, by use of a hardened steel die and hydraulic press. Active material is then removed from the edge of the cathode sleeve by rubbing with aluminium-oxide paper. The most important step in the processing of the cathode is sintering: *see* below. After sintering, the cathodes are stored in evacuated ampules until ready for use.'

The paper describes the test methods used in evaluation of moulded cathodes and reports results of the examination of the effects, on emission and life, of variations in nickel powder, alkaline-earth carbonates, and reducing agents, and the influence of sintering and ageing conditions. The test diode used for the emission tests is illustrated.

All the cathodes were life-tested for 1000 hours at a cathode temperature of 850°C. (brightness) and a plate voltage of 50v. (A figure in the paper illustrates typical current densities during 1000 hours of life.) The life test was interrupted periodically and the pulse emission of the cathodes was measured at 850°C., a peak plate voltage of 1000 v., a pulse length of 2·5μ/sec. and a repetition rate of 60 pulses/sec. (A figure shows peak current density as a function of peak-plate voltage.)

Although the study of cathode-pellet materials was not exhaustive, various compositions were tested, with the following resultant conclusions:

'The most useful emissive material is a triple carbonate containing 57 wt.% barium carbonate, 39% strontium carbonate, and 4% calcium carbonate. Cathode pellets were made also from a triple carbonate containing 13% calcium carbonate, and from barium carbonate alone. Satisfactory emission was obtained from all the cathode pellets, but those containing barium carbonate alone were somewhat gassy.

'Three nickel powders having particles of different sizes were used: Type B carbonyl nickel (1–3μ), standard carbonyl nickel (9–14μ) and Sherritt Gordon nickel powder (60μ). Although the emission from cathodes using Type B material was best, the difference was not necessarily significant.

'Three reducing agents were tested as activators: zirconium hydride, carbon, and titanium hydride. Carbon is gassy and results in short-lived emission. Zirconium hydride gives satisfactory results. Titanium hydride is satisfactory, but has no particular advantage over zirconium hydride.'

Sintering is the process which is of major importance in the processing of moulded nickel cathodes. During this process the cathodes are held in a nickel boat within a 'Vycor' tube which, in turn, is in a muffle furnace. Nitrogen and hydrogen are passed in series through a deoxidizer, an electro drier, a liquid-nitrogen trap, and finally the 'Vycor' tube. The hydrogen flows at the rate of 200 cm.^{3}/sec., the nitrogen at 50 cm.^{3}/sec. Details are given of the time-temperature schedule, which is critical. A figure showing pulse current density (measured periodically during life) as a function of hours of life demonstrates the improvement resulting from sintering. (The mechanism of the sintering process is not yet fully understood, but the authors make some suggestions as to the reactions occurring.) Data on pulse emission as a function of life are plotted

for cathodes (1) un-aged, (2) aged for 15 minutes, and (3) aged for 30 minutes. In these tests the best performance was obtained with the 15-minute treatment, but it is pointed out that uncontrollable variations in construction and processing of cathodes may require different ageing times to secure optimum results.

Moulded nickel cathodes have been used to advantage in several practical applications: they are particularly well suited for those involving exposure to air (as in demountable systems), or where the requirement is for delivery of large amounts of current in the presence of positive-ion bombardment, poor vacuum or a large electric field. Three typical examples of successful use are described.

Determination of the Die-Filling Characteristics of Nickel Powders

See abstract on p. 313.

Nickel in Coinage

E. F. FEELY: 'Recent Trends in Coinage Metals.'
Reprint from *Numismatist*, July, 1958; 5 pp.

By 1961 world production of nickel is expected to reach 300,000 metric tons per annum, a figure representing an increase of 130 per cent. over 1951. In the minting of coins the limited supply of nickel in the past few years has led inevitably to substitution, for nickel, of other more readily available metals, although in many cases they were less suitable for use in coinage. Exploitation of new deposits of nickel has recently not only increased production sufficiently to satisfy the demands of all consumers, but has also ensured stability of supply for many years to come. The possibility now exists, therefore, of a return to unrestricted use of nickel and nickel alloys for subsidiary and fractional coinage. With this possibility in mind, the author gives a review, based on figures for 1955 (the latest year for which data are available), of world consumption of metals and alloys used in coinage.

Statistics are given for eleven metals, which, in descending order of the tonnage used for this purpose, are: copper, nickel, silver, aluminium, steel, iron, zinc, tin, magnesium, manganese and gold. Only nickel, aluminium, zinc and iron were used unalloyed for coinage, and of these nickel alone has proved really suitable. Notes are given on the countries in which coins of these metals were used, and on quantities. Other brief sections are concerned with alloy coinage (including cupro-nickel), the declining rôle of silver in this field, and stainless- or plated-steel coinage.

The paper includes a table giving details of world consumption of nickel in nickel, cupro-nickel, silver, nickel-brass and nickel silver coinage, in the years 1953, 1954 and 1955, together with the total tonnages used in the various coins up to and including 1955. Total use of nickel in coinage from 1850 to 1955 was 65,646 tons (66,171,702 kg.).

Welding of Nickel

See abstract on p. 351.

Determination of Nickel in Soils and Rocks

R. E. STANTON and J. A. COOPE: 'Modified Field Test for the Determination of Small Amounts of Nickel in Soils and Rocks.'
Trans. Inst. Mining and Metallurgy, vol. 68, pp. 9–14; *Bull. Inst. Mining and Metallurgy*, 1958, No. 623.

The salient features of methods described in the literature for determination of nickel in soils and rocks are briefly reviewed, with an indication of the degree of sensitivity achieved by the respective techniques.

The method described by the author is a modification based on procedures referred to in two unpublished theses.

According to the modified method, a nickel-furildioxime complex extracted with benzene is used in colorimetric comparison with a series of standards similarly prepared. Analytical procedure is detailed and particulars are given of apparatus required for application of the method in the field.

It is claimed that nickel content of soil and rock samples can be determined by this technique in the concentration range 1–15,000 p.p.m. About 80–1000 determinations can be made per man-day, and the accuracy is of the order of ± 25 per cent. It is stated that extensive field tests in Africa have shown that the method can be used satisfactorily in geochemical prospecting.

Determination of Traces of Nickel in Crops and Foodstuffs

W. D. DUFFIELD: 'A System for the Determination of Certain Trace Metals in Crops.'
Analyst, 1958, vol. 83, Sept., pp. 503–8.

In the system described, which is suitable for rapid routine determinations, copper, cobalt, nickel, molybdenum and boron are determined by paper chromatography. Zinc and manganese are estimated colorimetrically, and, in certain cases, a colorimetric procedure may be used for molybdenum. Precise details of technique are given, and a report is made of check recovery experiments in which known amounts of various metallic salts were added to the hydrochloric-acid solution of the ash.

Separation of Cobalt and Nickel as Pyridine Thiocyanates

J. H. W. FORSYTHE, R. J. MAGEE and C. L. WILSON: 'The Analytical Chemistry of the Pyridine Thiocyanates. I. The Separation of Cobalt and Nickel by Solvent Extraction.'
Talanta, 1958, vol. 1, Sept., pp. 249–51.

Conditions under which the pyridine thiocyanates of cobalt and nickel form were studied. Both compounds are soluble in organic solvents and the process described in this paper is based on solvent extraction. After separation, the metals are determined colorimetrically in the extracts.

ELECTRODEPOSITION AND OTHER COATING METHODS

Watts Nickel-Plating Solutions: Composition and Properties

E. B. SAUBESTRE: 'The Chemistry of Watts Nickel-Plating Solutions.'

Plating, 1958, vol. 45, Sept., pp. 927–36.

This paper makes a comprehensive evaluation of the Watts nickel-plating solution, by discussion of the nature and effect of each of its major constituents. For the purpose of calculation, the following formula has been arbitrarily chosen as typical:

	g./L.
$NiSO_4.6H_2O$	350
$NiCl_2.6H_2O$	60
H_3BO_3	30
pH	2·5
Temperature (°C.)	50
Current density (amp./sq. ft.) ..	50

It is shown that—

Nickel Sulphate determines the cathode reduction conditions. The deposition of nickel is determined from the standard potential, as corrected for the activity of the nickel ion in a sulphate bath, and the activation overvoltage of nickel. Factors which make the deposition of nickel reversible are considered.

Nickel Chloride is considered in terms of anode dissolution and its rôle in influencing high-speed deposition at the cathode. It is demonstrated that, at most current densities, nickel chloride has little effect in a Watts bath.

Boric Acid, as buffer and complexing agent, is discussed in terms of the various polymers which form in solution from boric acid. Brief reference is made also to other buffers.

Anti-Pitting Agents used in U.S.A. and in Europe are described, with details of composition, and discussion of the nature of their action. In this connexion reference is made to hydrogen peroxide, peroxysulphates, peroxyborates, nitrates and various wetting agents.

An appendix to the review contains (1) notes on the activity of water, as affecting aqueous plating solutions; (2) experimental data obtained in working all-sulphate solutions, with special reference to definition of the rôle of the chloride ion at the cathode, and the use of insoluble anodes in a solution containing ion-exchange membranes.

Behaviour of Various Grades of Nickel Anode in Nickel-Plating Solutions

E. RAUB and A. DISAM: 'The Anodic Behaviour of Various Types of Nickel in Nickel-Plating Solutions.'

Metalloberfläche, 1958, vol. 12, June, pp. 161–7; July, pp. 193–7.

Wesley, in a fundamental study of anodic dissolution of nickel, has interpreted the mechanism in terms of difference in the electronic structure of crystalline nickel and the nickel ion. The behaviour of various anodes was investigated, with particular reference to sludge formation, by Offermanns *et al.* The findings of all these authors (to which reference was made in *Nickel Bulletin*, 1956, vol. 29, No. 10–11, p. 182; *ibid.*, 1957, vol. 30, No. 7–8, p. 111) are summarized in the present paper by way of introduction to the work described. The investigation was carried out with the aim of obtaining additional information on the behaviour of nickel anodes in matt- and bright-nickel-plating solutions.

The following five grades of anode were studied:

(1) and (2) High-purity rolled anodes with a minimum nickel content of 99·8 per cent. Both anodes contained trace amounts of cobalt, copper, iron, manganese, magnesium, silicon and lead. Anode (2) had a carbon content of 0·02 per cent. and considerably higher magnesium and manganese contents than anode (1); its iron and silicon contents, were, however, lower.

(3) An electrolytic anode, the impurity content of which was considerably lower than that of (1) or (2).

(4) A depolarized rolled anode containing an addition of 0·4 per cent. of nickel oxide.

(5) A rolled anode with controlled additions of silicon (0·34 per cent.) and carbon (0·2 per cent.). These elements, by formation of a film of graphite and silicon dioxide on the anode, are intended to obviate the need for an anode bag.

The paper includes microphotographs which illustrate the structure of each anode.

Four nickel-plating solutions were used in the investigation: two of Watts type, a straight-chloride solution, and a commercial bright-nickel-plating electrolyte of high levelling power. The solutions were operated at 20° or 50°C. and the pH was varied in the range 2–4.

Three variables were studied:

(*a*) Variation of anode potential with current density.

(*b*) Anode current efficiency.

(*c*) Sludge formation during anodic dissolution.

Sludge formation and current efficiency were investigated with only two of the electrolytes: the Watts solution, containing (g./L.) nickel sulphate 250, nickel chloride 40, boric acid 30; and the bright-nickel-plating solution.

Anode-potential/current-density curves were obtained (and are presented) for each of the five anodes, in each of the four electrolytes. Current efficiency and degree of sludge formation (the latter as a percentage of the dissolved nickel) were determined using current densities in the range 1–5 amp./dm.^{2}: the data derived are presented in tabular form.

The behaviour of the nickel anodes in the various solutions used is discussed in detail by the authors. Their main findings are outlined below.

The rolled high-purity anodes (1) and (2) were found to exhibit the highest degree of polarization. The curves for the electrolytic (3) and depolarized (4) anodes proved to be virtually identical: polarization was low and that of anode (3) was, under certain conditions, the lower of the two. The anode containing carbon and silicon (5) dissolved with a polarization which lay somewhere between those of anodes (1) and (2) and that of anode (4). With all five anodes polarization was at a minimum during deposition from the straight-chloride solution; the highest values determined were with the bright-nickel electrolyte.

Current efficiencies of all the anodes were in the region of 100 per cent.

The quantity of sludge produced varied widely with the individual anode. Sludge formation with the depolarized anode, which was particularly intensive, is attributed to the eutectic arrangement of the nickel oxide in the nickel lattice. Little difference was discerned between anodes (1) and (2) in this respect, while with the alloyed anode (5) sludge formed only when the graphite-rich anode film was imperfectly formed. Minimum formation of sludge was observed with the electrolytic anode.

Under certain operational conditions anodic attack on anodes (1) and (2) occurred in the form of pitting, and the resultant sludge was relatively coarse-grained and metallic; suitable control of the conditions was found, however, to produce, on anode (2), a film which proved beneficial in lessening sludge formation. In the case of the electrolytic anode, attack progressed uniformly and the usual bud-like surface structure was observed: the small amount of sludge formed was very fine-grained. (Emphasis is, however, placed on the wide variation in behaviour which such anodes show in service.) The depolarized anode was also corroded uniformly and, depending on the operational conditions, appeared grey or metallic under the surrounding sludge: the sludge contained in the solution was metallic and relatively fine-grained. Anode (5) corroded uniformly except when the surface film was incomplete: pitting then occurred. Where sludge did form it was very fine-grained and had a black non-metallic appearance.

Electrodeposition of Metals from Sulphamate Solutions

T. L. R. CHAR: 'Electroplating and Electrorefining of Metals from the Sulphamate Bath.'

Electroplating and Metal Finishing, 1958, vol. 9, Oct., pp. 343–6.

This article comprises a review of recent work on electrodeposition from sulphamate solutions, including the results of investigations carried out in the author's laboratory. It is documented by a *bibliography* of 34 items.

A section of the review is devoted to electrodeposition of nickel from sulphamate solutions, and, in this connexion, a tabular summary is made of the composition of electrolytes used by various investigators and of operating conditions.

Electrodeposition of Nickel on Group IVB-VIB Metals

The following abstract is published of a paper recently presented before The Electrochemical Society:

E. B. SAUBESTRE: 'Electroplating on Group IVB-VIB Metals.'

Jnl. Electrochemical Soc., 1958, vol. 105, Aug., p. 160C.

'In a new plating method, the conventional etching step is eliminated. Instead, the substrate is treated cathodically to form a hydride-covered surface, which permits good adhesion of thin nickel deposits. The hydride is then removed by a vacuum bake, followed by further plating as desired. It was found possible to plate over oxide-coated Nb and Nb-Ti alloys, without need for activation or post-plating baking operations. The oxide was applied by anodizing.'

Corrosion-Resistance of Multi-Layer Nickel Coatings

'Multilayer Nickel Coatings.'

Metal Progress, 1958, vol. 73, June, pp. 95–6.

In 1947 the American Society for Testing Materials initiated a programme of atmospheric exposure tests, with the primary aim of studying the relative corrosion-resistance of copper/nickel/chromium and nickel/chromium decorative coatings, on steel of the type used for car bumpers. Some of the specimens were coated with two layers of nickel from a Watts solution: the first layer was buffed and on it a second identical coating was applied. It was found that considerably higher protection was conferred by these coatings than by a single nickel coating of equivalent thickness or by a copper/nickel coating. (A detailed abstract of the reports on these tests appeared in *Nickel Bulletin*, 1957, vol. 30, No. 6, p. 86: see also *ibid.*, No. 7–8, p. 112.) Commercial bright-nickel-plating techniques have been modified in the light of these findings, and some indication of the results achieved is given in this summary of a paper presented by M. B. DIGGIN to the Cleveland Branch of the American Electroplaters' Society.

Double-layer coatings, produced by deposition of a semi-bright undercoating of nickel (about 80 per cent. of the total thickness) followed by a fully-bright nickel coating, on copper-plated zinc die-castings, have exhibited, in atmospheric tests, a corrosion-resistance up to 30 per cent. higher than that of copper/nickel deposits of similar thickness. Photomicrographs are used to illustrate that in the duplex coatings corrosion starts in the top coating and them

instead of penetrating the undercoating, proceeds laterally along the top layer. This phenomenon is attributed to differences in the potentials of the two coatings, which lead to sacrificial action by the top layer, so protecting the underlayer.

Special Applications of Nickel Plating

H. C. CASTELL: 'Nickel Plating in Special Applications.' *Engineering*, 1958, vol. 186, Oct. 31, pp. 574–6.

Also available as *Publication* 1562 issued by THE MOND NICKEL CO., LTD.

Nickel plating has long been associated with its many decorative uses, but modifications of old, and development of new, plating methods have greatly extended the range of its applications. In this article the author emphasizes the versatility of present-day nickel-plating techniques, by describing some of the special applications which they have found in industry.

Salvaging of worn components by heavy nickel plating has become a regular practice in industry: the London Transport Executive makes wide use of this technique to reclaim such components as hubs, shafts, fluid fly-wheel housings, and dynamo armatures. Heavy nickel plating is used also to confer resistance to wear and corrosion on plant which is subjected in service to severe abrasive conditions (e.g., hydraulic rams and presses, and machinery used in the paper-making and printing industries).

The ability of electrodeposited nickel to reproduce, in exact detail, the contours of the surface being plated has paved the way for development of the many special applications of electroforming. The advantages of this technique are described, in connexion with its use in forming the master disc in the manufacture of gramophone records, and the master die for mass production of articles in the plastics industry. Details are also given of procedures employed to obtain complicated electroformed screens and grids of very fine mesh, which are used, for example, as filters in the food-processing industries, as sieves for powders and in paper-making, and as grids and screens in cathode-ray tubes.

The abrasion- and corrosion-resistant applications of electrodeposited nickel are exemplified by references to the protection such coatings confer on aeroplane propellers, plates used in letter-printing, meat-slicing and food-packing machinery, and cooking utensils.

A tin-nickel alloy can now be deposited from a fluoride solution, to give semi-bright coatings with a hardness in the region of 700 D.P.N. and with a low coefficient of friction which makes the coating ideal for instrument parts. A nickel-phosphorus-alloy coating deposited by the electroless chemical-reduction process may be heat-treated to a hardness of about 1000 D.P.N.; use of the electroless-plating technique also ensures perfectly uniform deposition over complex surfaces. Some applications of the process are mentioned.

Nickel Plating prior to Enamelling

'Hotpoint Lowers Porcelain Enamel Firing Temperatures.'

International Enamelist, 1958, vol. 8, No. 2, pp. 28–9.

In a review of improved methods which have been introduced into the process used for enamelling 'Hotpoint' range bodies, reference is made to the fact that the thickness of nickel undercoat standard in these works has been raised from 0·010 to about 0·040 gram. per sq. ft.

Copper De-greasing of Components prior to Nickel Plating

'Copper Degreasing before Bright-Nickel Plating.'

Electroplating and Metal Finishing, 1958, vol. 11, Sept., p. 325.

The article outlines the salient features of a combined copper-flash/de-greasing process developed by A. Reymond et Cie., Switzerland. The process, originally intended for treatment of brass bars prior to bright-nickel plating, has since been successfully applied to de-greasing of other materials, e.g., copper and iron and zinc-base die castings. Brief notes are given on the solutions and procedures used.

'Micrograin' Nickel Linings for Propeller Blades

'The Development of 'Micrograin' Nickel.'

Corrosion Prevention and Control, 1958, vol. 5, Aug., pp. 56–8.

When details were first given of the development of the 'Micrograin' process for electrodeposition of nickel (see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 6, p. 165), deposits produced by the technique were expected to find a wide range of applications as electroforms. Such electroforms are now under test as linings for the leading edges of aircraft propellers. The lining is cemented, by means of epoxy adhesive, over the delicate rubber-embedded de-icer elements of the propeller, so providing a sheath which protects these components from damage by hail, or by stones thrown up during take-off or landing.

Tests conducted on propeller blades for 'Viscount' aircraft have confirmed the suitability of 'Micrograin' nickel for this purpose. Pebbles of various sizes were directed into blades operating at high speed, and the performance of a 'Micrograin' sheath was compared with that of one made from stainless steel of equal weight. After four test runs the stainless-steel lining was crushed; the 'Micrograin' electroform showed some signs of wear, but the elements were undamaged. The condition of both sheaths after two and four test runs is illustrated.

Despite its extreme hardness, 'Micrograin' nickel may be bent easily without fear of cracking, and shapes with re-entrant surfaces may be electroformed and then closely fitted to the finished product: advantage is taken of this characteristic to produce a jet-engine circular intake cowl which tapers

towards the inside. Other applications for which 'Micrograin' nickel is expected to prove suitable include linings for boat propellers, counter tops and car bumpers.

Electrodeposited Porous Nickel for Battery Plates

The following abstract is published of a paper recently presented before The Electrochemical Society:

J. J. MACDONALD, D. G. GAGE and E. C. BROWN: 'Electrodeposition of Porous Nickel.'

Jnl. Electrochemical Soc., 1958, vol. 105, Aug., p. 160C.

'In an attempt to simplify nickel–cadmium-battery production, studies of the electroforming of porous nickel were undertaken with a view to replacing the normal sintered plaques. Plaques with up to 80 per cent. porosity have been electrodeposited reproducibly from a Watts-type bath to which has been added lamp black or graphite. When tested as battery positives, the best plaques showed capacities 70 per cent. as high as those of sintered nickel plaques.'

Electrodeposition of Nickel-Aluminium Alloys

The following abstract is published of a paper recently presented before The Electrochemical Society:

D. COUCH and J. CONNOR: 'Nickel–Aluminium Alloys Formed by Electrodeposition.'

Jnl. Electrochemical Soc., 1958, vol. 105, Aug., p. 160C.

'Nickel–aluminium alloy coatings were produced by the electrodeposition of aluminium over electrodeposited nickel. The basis metal was steel. The aluminium was plated from fused salt baths operated at 170°–1000°C. and from an organic bath operated at room temperature. The coatings had a hardness of about 700 Vickers and were much superior to nickel coatings for the protection of steel in salt-spray tests. The oxidation of these alloys at 1000°C. was much slower than that of pure nickel. Attempts to co-deposit nickel and aluminium from an organic type of bath were not successful.'

NON-FERROUS ALLOYS

Research at The Birmingham Laboratory of The Mond Nickel Company, Ltd.

See abstract on p. 330.

Hereford Works of Henry Wiggin and Company, Ltd.

'Expansion of the Hereford Works of Henry Wiggin.'

Machinery (*Lond.*), 1958, vol. 93, Oct. 8, pp. 851–2.

See also *Metal Industry*, 1958, vol. 93, Oct. 3, pp. 279–82.

With the aim of concentrating all facilities for the production of nickel-base alloys in one plant, Henry Wiggin and Company, Ltd. have embarked on a long-term programme of expansion and modernization at their Hereford works. By 1965 the area covered by the works will have been expanded from 7 acres to 25 acres, and the production stages carried out at the Company's Birmingham and Glasgow works will all have been incorporated in the Hereford plant. The major melting processes have already been transferred from Birmingham to Hereford. These articles give a brief general outline of the plant used in the melting, casting, machining, extrusion and rolling sections of the works, and of the facilities available in the metallurgical laboratories.

Separation of Cobalt and Nickel as Pyridine Thiocyanates

See abstract on p. 316.

Influence of Nickel on Properties of Gunmetal Castings

F. HUDSON, D. R. WOOD and J. F. GREGG: 'Properties of Copper-base Alloy Castings.'

Brit. Foundryman, 1958, vol. 51, Oct., pp. 469–81; disc., pp. 482–5.

Reprint available as *Publication* 1577, issued by THE MOND NICKEL CO., LTD.

Although information on the influence of section-thickness on the properties of castings in copper-base alloys is relatively sparse, results obtained from the few investigations carried out in this connexion have shown conclusively that mechanical properties are affected by variation in section thickness. In a review of the literature, the authors give a brief résumé of the findings of REICHENECKER (*Materials and Methods*, 1953, vol. 38, July, pp. 80–1) and other authors. Reichenecker used a step-bar having a section varying from $\frac{3}{4}$ to 2 in. (9–50 mm.) in thickness, and preliminary tests in the laboratories of The Mond Nickel Company, using a similar type of step-bar, gave results (on gunmetals) which confirmed those obtained by Reichenecker, and also indicated the beneficial effect of nickel additions (see *Trans. Inst. Marine Engineers*, 1954, vol. 66, p. 169). A more detailed investigation of the influence of casting section on the properties of gunmetals, with or without addition of nickel, is reported in the present paper.

Two leaded gunmetals (copper–tin–zinc–lead alloys of 85-5-5-5 and 86-7-5-2 types) and an 88-10-2 copper–tin–zinc alloy were selected for study. To determine the effect of nickel additions, tests were also made on specimens of the two leaded gunmetals with additions of up to 4 per cent. of nickel. Tensile and Izod-impact properties were determined, and density and hardness were also measured.

Results are presented *in extenso*, in tabular and graphical form, and micro- and macrographs are used to indicate structural variation with thickness. Comparative data are given for aluminium bronze and high-tensile brass.

Composition was found to have an important bearing on the sensitivity of the gunmetals to variation in section-thickness. Values for room-temperature tensile strength and elongation of all three alloys

fell markedly with increase in section, and the degree to which they fell increased with decrease in tin content and increase in lead content.

With respect to the 85-5-5-5 gunmetal, the mechanical properties of thick sections were seriously reduced by the presence of massive grain-boundary segregates, in addition to a variable amount of porosity. A step-bar casting of an alloy of the same type, but containing 2 per cent. nickel and 4·2 per cent. tin, was, however, found to be substantially free from micro-porosity. Addition of nickel resulted in a finer and more uniform dispersion of the lead particles and a higher and much more uniform level of properties in castings of varying section. A larger addition of nickel (4·2 per cent.), in the presence of 4·5 per cent. tin, was shown to have a slightly beneficial effect on tensile strength, but was deleterious with respect to percentage elongation and impact strength.

The authors draw the following general conclusions:

'From the information available it would appear that alloys with a short freezing range, such as aluminium bronze and high-tensile brass, are not unduly sensitive to the effect of section-thickness, and castings can be produced in these alloys with a high level of mechanical properties, approaching those obtainable from separately cast test-bars. In the case of alloys with a long freezing range, such as tin bronze and gunmetal, the properties of castings may vary widely with section thickness and may be unrelated to those obtained from separately cast test-bars.

'All the standard gunmetals in use to-day have been evolved by trial and error and, as a result of systematic study, compositions may have to be modified to obtain an improvement in casting quality. From a study of the data given in this paper, it is evident that none of the existing standard gunmetals is capable of providing the ideal combination of properties needed for the production of quality castings, namely a uniformly high level of strength, ductility, and pressure-tightness in castings of variable section.

'It can be postulated that an alloy containing about 6-7 per cent. Sn, 3-4 per cent. Zn, 3-4 per cent. Pb, 1½-2½ per cent. Ni, balance copper, may provide this ideal. Such an alloy would be easy to handle in the foundry and would be better suited for the production of quality castings than any of the standard gunmetals in use today. It could probably replace the 88-10-2, 86-7-5-2 and 85-5-5-5 alloys. Gunmetal castings in this modified composition would be less sensitive to the effect of casting section and, if properly made, should have a tensile strength of not less than 16 tons/sq. in. (25 kg./mm.²). They will be just as adaptable as 85-5-5-5 gunmetal to the production of pressure-tight castings, and they are likely to have improved properties at elevated temperatures. Furthermore, the use of such an alloy would undoubtedly enable castings to be more effectively designed, particularly as regards the use of thinner sections, and this could result in a considerable saving in weight. In the valve industry alone this might lead to substantial savings.'

Diffusion in the Aluminium-Nickel System

L. S. CASTLEMAN and L. SEIGLE: 'Fundamentals of Diffusion Bonding. II.'

U.S. Atomic Energy Commission, Report SEP 245, Feb. 15, 1958; 24 pp.

Investigation of bond formation in the cladding of fuel elements by pressure welding has shown that the size and nature of the diffusion zone are affected by variations in applied pressure. Increasing pressure alters the thicknesses of intermediate phase layers in the diffusion zone and changes the strength of the bond.

The report of *Part I* of this investigation dealt with a study of the literature relating to phenomena believed to have a bearing on the subject: (1) the kinetics of formation of intermediate phases, and (2) the effect of stress on rates of diffusion.

As a result of that survey, the aluminium-nickel system was selected for initial experimental investigation. Work done prior to June 1956, and recorded in Part I of the report, is summarized in this later report, which covers the period to June 1957.

It is concluded that

'(1) Diffusional bonding in the Al-Ni system, in the temperature range 400°-625°C., occurs by formation and growth of the Ni_2Al_3 (gamma) and the $NiAl_3$ (beta) layers. Other phases are either not present or occur in sub-microscopically thin layers.

'(2) The growth of the gamma layer in the temperature range 400°-625°C. is controlled by volume diffusion in the gamma phase, once a minimum layer thickness of the order 0·025-0·030 mm. is reached. Prior to this, the layer grows more rapidly than volume diffusion considerations would predict.

'(3) Tentatively, the chemical diffusivity in the gamma phase has been calculated to be of the order of $0{\cdot}7 \times 10^{-10}$ cm.²/sec. at 600°C.; the heat of activation is in the vicinity of 32,000 cal./mol. over the temperature range 400°-625°C.

'(4) The beta phase grows at a much less rapid rate than does the gamma phase; also, the growth of the former is relatively temperature-insensitive in the range 400°-625°C.

'(5) Pressure decreases the rate of growth of the gamma layer; tentatively, it appears that one effect of pressure is to lower the chemical diffusivity in the gamma phase; it is estimated that a pressure of 5 tons/sq. in. (8 kg./mm.²) lowers the diffusion coefficient by 33 per cent. at 600°C.'

In the later investigations additional information has been obtained with regard to the growth of the beta phase, and a method has been developed by which the kinetics of the growth of that phase can be studied.

Work has been begun on the kinetics of interface movement and layer growth in systems which have less complicated phase-equilibrium relationships

than those existing in the aluminium-nickel system, e.g., antimony-tin, aluminium-antimony and copper-silver.

Solid Solubility of Phosphorus in Nickel

See abstract on p. 314.

Nickel-Aluminium-Bronze Marine Propellers

F. HUDSON: 'The Development of High-Tensile Aluminium-Bronze Alloys for Marine Propellers in Great Britain and the United States of America.'

Amplified Reprint from *Shipbuilder and Marine Engine-Builder*, 1957, vol. 64, Dec., pp. 681–7.

(See abstract in *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 46.)

MOND NICKEL CO., LTD., *Publn.* 1430*; 25 pp.

Nickel-Chromium Electrical-Resistance Alloys

See abstract on p. 340.

Nickel-Alloy Coinage

See abstract on p. 316

Welding of High-Nickel Alloys

See abstract on p. 351.

Welding of Dissimilar Metals: Technique, Electrodes and Welding Wire

See abstracts on p. 350.

NICKEL-IRON ALLOYS

Hall Effect in Iron-Nickel Alloys

N. V. BAZHANOVA: 'On the Hall Effect in Ferromagnetics.'

Soviet Physics JETP, 1958, vol. 6(33), Mar., pp. 440–3. Translated from *Jnl. Experimental and Theoretical Physics* (*U.S.S.R.*), 1957, vol. 33, Sept., pp. 567–70.

In the work described the author studied the Hall effect in iron-nickel alloys of the 'Invar' series, in the region of the Curie temperature.

It is concluded that the Hall effect in ferromagnetic materials in strong fields (above technical saturation) is not determined by the magnetic field, but increases linearly with increase in true magnetization. It should therefore be expressed in terms of the following equation, developed by Volkov:

$$E = R_t I_t + R_i I_i,$$

where I_t is the magnetization of the ferromagnetic, R_t is the constant of the so-called extraordinary Hall effect, R_i is a constant of the material describing the Hall effect in magnetic fields in which it is determined by the true magnetization, and I_i is the true magnetization.

A relationship between the Hall e.m.f. and the magnetic field has been confirmed by experimental results.

* *We shall be pleased to supply a free copy of this publication.*

CAST IRON

Cast Iron Handbook

GRAY IRON FOUNDERS' SOC., INC.: 'The Gray Iron Casting Handbook.'

Edited by C. F. WALTON.

Published by *Gray Iron Founders' Soc.*, 1958; 607 pp.

This handbook is probably the most complete manual on iron castings yet published, in that it represents a correlation of information selected from thousands of scientific papers, lectures and articles which have recorded the technological accomplishments and progress made during many years of research and process development by the grey-iron industry.

The scope of the book is indicated by the chapter headings given below.

Advantages of Iron Castings

Design of Gray Iron Castings

Specifying and Purchasing

Properties, covering grey, nodular (S.G.), white and alloyed cast irons, and a discussion of microstructure, composition and section-size in relation to mechanical properties in castings of the respective materials. The review covers properties at both normal and elevated temperatures. Corrosion-resisting characteristics are also considered.

Heat-Treatment of Gray Iron. A comprehensive discussion, on the basis of theory and practical experience.

Welding, Joining and Cutting of Gray Iron

Machining and Grinding

} Both sections embody information collected from the somewhat scattered literature, together with specific details of recommended practice.

Metallic and Non-Metallic Coatings for Gray Cast Iron

Each chapter is supported by a *bibliography* of authoritative literature, and the value of the manual is enhanced by a useful glossary of terms used in the foundry industry.

Engineering Properties of Austenitic Spheroidal-Graphite Cast Irons

INTERNATIONAL NICKEL CO., INC.: 'Engineering Properties of 'Ni-Resist' Ductile Irons.'

Second Edn., issued 1958; 28 pp.

The brochure contains a comprehensive summary of engineering data on austenitic cast irons of the spheroidal-graphite type, containing from 18 to 36 per cent. of nickel. S.G. 'Ni-Resist' cast irons offer a combination of advantages: those derived from their

spheroidal-graphite structure (tensile strengths ranging from 55,000 to 88,000 p.s.i. (24·5–39·5 t.s.i.; 38·5–70 kg./mm.²), with elongations of 4–40 per cent.) and those resulting from their high alloy content. Five grades of the material are available, the compositions of which are designed to give properties suitable for specific applications. Characteristic of the wide range of properties obtainable in these grades are castability; machinability; resistance to corrosion by acids, alkalis and salts; toughness, comparable at ordinary temperatures to that of steel, and well retained at low temperatures; resistance to erosion and frictional wear and to heat and oxidation; thermal-expansion characteristics which can be varied, as desired, by selection of suitable nickel contents. Some of the irons are non-magnetic, which is an added advantage for certain applications in the electrical and allied fields.

The information in the publication is presented in sections relating to: production, machining and welding; mechanical and physical properties; strength properties; heat-treatment; erosion-resistance; corrosion-resistance; high-temperature properties; thermal expansion and conductivity; low-temperature properties; commercial applications.

CONSTRUCTIONAL STEELS

Austenite Transformation in Nickel-Alloy Steel of Eutectoid Carbon Content

R. A. GRANGE, P. T. KILHEFNER and T. P. BITTNER: 'Austenite Transformation and Incubation in an Alloy Steel of Eutectoid Carbon Content.'
Amer. Soc. Metals, Preprint 93, Oct. 1958; 19 pp.

Transformation of austenite, as occurring isothermally and on continuous cooling, was studied in a 1 per cent. nickel, 1 per cent. chromium, 0·26 per cent. molybdenum steel at a carbon level (0·57%) such that neither pro-eutectoid ferrite nor pro-eutectoid carbide was formed. Comparison of the resulting T.T.T. diagrams shows that the methods which have been proposed for correlating the two groups of results are unsatisfactory for this type of steel, due to the fact that holding metastable austenite in certain temperature ranges (whether accomplished by isothermal treatment or by comparatively slow continuous cooling) retards subsequent transformation at a lower temperature. In particular, exposure of metastable austenite in the 1000°–900°F. (540°–480°C.) range markedly retards subsequent transformation. The discrepancy between measured and derived cooling transformation diagrams is explicable in large measure by the austenite-stabilizing effect of the slower cooling, through 1000°–900°F. (540°–480°C.), of specimens used in measuring cooling transformation, as compared with the rapid cooling of specimens used in measuring isothermal transformation.

Transformation Characteristics of Nickel-Alloy Steels

MOND NICKEL CO., LTD.: 'Transformation Characteristics of Direct-Hardening Nickel-Alloy Steels.' *Publn.* 1268.*
Revised and Amplified Edn., published 1958; 91 pp.

The first Atlas of Isothermal-Transformation Diagrams of Nickel Steels, published in 1950, presented isothermal data for thirteen nickel-containing B.S. En steels of commercial manufacture. In 1952 this Atlas was reviewed, and extended to include end-quench hardenability data for the same series of steels. The information given in these two earlier editions demonstrated the value of isothermal-transformation studies as an aid in the understanding of steel structures, and drew attention to the influence of structure on mechanical properties. Special heat-treatments based on isothermal-transformation diagrams were also described, and attention was drawn to the limitations inherent in diagrams of that type.

One of the difficulties of applying isothermal data to practical heat-treatments in which a steel is continuously cooled from the austenitizing temperature is that it is not possible to assess, from isothermal-transformation diagrams, the extent and type of transformations which occur during cooling. In view of this difficulty, the Atlas has again been revised, and in this third edition has been amplified to include transformation diagrams for both continuous-cooling and isothermal conditions. The continuous-cooling diagrams, presented in the form considered to be most suitable for practical purposes, cover the range of cooling rates involved in oil-quenching 1-in. to 6-in. diameter bars, i.e. the rates most usually occurring in industrial operations.

The scope of the information contained in the publication is indicated below.

Isothermal-Transformation Diagrams.

This chapter describes methods for the determination and presentation of such diagrams, and includes discussion of the effect of composition on isothermal-transformation characteristics, the martensite reaction, and the occurrence of retained austenite in heat-treated steels.

Influence of Structure on Mechanical Properties

Special Heat-Treatments based on Isothermal-Transformation Diagrams

This section deals with martempering, interrupted quenching, and time-quenching, austempering, and isothermal annealing, and with results obtained by such treatments.

Continuous-Cooling Transformation Diagrams: Determination and Presentation.

* A copy of this publication will be sent to metallurgists and engineers in whose work it may prove useful. It is requested that in all cases the interest of the writer be indicated, and where possible, the use of the firm's notepaper will be helpful in assisting further technical contacts.

Limitations of Transformation Diagrams

Hardenability

Discussion of the influence of chemical composition; measurement of hardenability; applications and limitations of end-quench hardenability curves.

Effects of Tempering

A review of the characteristics of the martensite decomposition during tempering, and associated effects; also the nature of carbides in tempered alloy steels.

The second section of the book comprises a series of charts showing transformation and hardenability data relating to the following types of steel:

B.S. En 12	1% Ni Steel
B.S. En 22	3½% Ni Steel
B.S. En 23	3% Ni-Cr Steel
B.S. En 24	1¼% Ni-Cr-Mo Steel
B.S. En 25	2½% Ni-Cr-Mo (Medium-Carbon) Steel
B.S. En 26	2½% Ni-Cr-Mo (High-Carbon) Steel
B.S. En 28	3½% Ni-Cr-Mo Steel
B.S. En 30A	4¼% Ni-Cr Steel
B.S. En 30B	4¼% Ni-Cr-Mo Steel
B.S. En 100	Low-Alloy Steel
B.S. En 110	Low Ni-Cr-Mo Steel
B.S. En 111	Low Ni-Cr Steel
B.S. En 160	2% Ni-Mo Steel

The information given in the text is supported by a selective *bibliography* of 86 references to technical literature.

Multiplication Factors for Calculation of Hardenability

C. F. JATCZAK and D. J. GIRARDI: 'Multiplying Factors for the Calculation of Hardenability of Hypereutectoid Steels Hardened from 1700°F. (927°C.).'

Amer. Soc. Metals, Preprint 99, Oct. 1958; 15 pp.

Hardenability effects of manganese, silicon, chromium, nickel and molybdenum, singly and in combination, were studied in 1 per cent. carbon steels by the end-quench test, at a quenching temperature of 1700°F. (927°C.), and from normalized and spheroidized prior structures. Hardenability factors were obtained for each element up to a level of 1·50 per cent. or above, with the exception of molybdenum, the effect of which was determined up to only 1·0 per cent.

Two different sets of hardenability factors were derived. One set, based on a normalized prior structure, can be used to predict the case hardenability of carburizing steels at carbon levels of 0·75–1·25 per cent.: the other, based on a spheroidized prior structure, is for prediction of hardenability in homogeneous hypereutectoid steels, such as tool steels.

Ultra-High-Tensile Steel : British Developments

L. MARSHALL: 'Ultra-High-Tensile Steel: Properties and Applications of a New British Material.'

Engineering, 1958, vol. 186, Oct. 3, pp. 446–8.

Interest in ultra-high-tensile structural steels for use in the aircraft industry and other spheres has led to investigations by British steelmakers of the basic metallurgical characteristics of such steels, their fabricating properties and the design considerations which will be involved in their use. This article describes work done on a typical steel which gives promise of meeting the specification 'ultra-high-tensile', without undue loss of ductility and toughness. It is designated 'Rex 539'.

An independent survey of relevant information, together with results of preliminary tests, led Messrs. Thos. Firth and John Brown, Limited, to select the composition shown below for manufacture of the production of a 10-ton cast which was to be broken down into bars and billets for production of tubes and forgings (the latter being intended for design studies in connexion with aircraft components).

As a preliminary step, examination was made of the tensile-properties/tempering-temperature and Izod-impact/tempering-temperature relationships for the new steel, in comparison with similar relationships exhibited by typical ultra-high-tensile steels developed in the U.S.A. and by 2½ per cent. nickel-chromium-molybdenum steel to *B.S.* S.96. The results demonstrate the superiority of the 'Rex 539' steel in terms of tensile and proof-stress values. Whereas over the 250°–400°C. tempering range there is the usual pronounced temper-brittle dip in the properties curve for the S.96 steel, the new steel gives a more uniform response to tempering, with regard to Izod values obtained over the whole range of tempering temperatures. Its superior tensile properties by comparison with the American steels are also shown, in that in 'Rex 539' it is possible to obtain high tensile strength without undue lowering of the tempering temperature. A tensile value of more than 120 t.s.i. (190 kg./mm.²) can be obtained by tempering at 350°C., and 100-ton (157 kg./mm.²) proof stress is still possible with a tempering temperature of 390°C. At 120 t.s.i. (190 kg./mm.²) the P.S./U.T.S. ratio is 0·86.

Particular attention is directed to the significance of the high tempering temperatures which can be used for 'Rex 539' steel. This feature is important from three aspects: (*a*) the usefulness for applications where high temperatures are encountered, as, for example, in high-speed aircraft subject to dynamic heating; (*b*) there is less likelihood of 'locked-up' stress remaining in complicated forgings; and (*c*) the

Composition of 'Rex 539' Steel

C %	Si %	Mn %	Ni %	Cr %	Mo %	V %
0·37	1·58	1·57	1·77	0·11	0·4	0·21

steel will be easier to handle from a practical heat-treatment aspect.

Machining experiments carried out on an axle made from 'Rex 539', heat-treated to the u.h.t. condition, indicate that the material can be handled without any revolutionary changes in machining technique, but that care will have to be taken to avoid cracking and scorching during grinding. Forgings selected for reproduction in 'Rex 539' were of the type in use for aircraft-undercarriage construction (now made from S.99 steel). Notes on fabrication technique include recommendation of a double softening treatment to ensure that the steel is in the 60–70 ton (95–110 kg.) condition for machining, and application of the heat-treatment normal for the steel just prior to grinding to final size.

Two leading manufacturers of tubes report that, by use of suitable techniques, no serious difficulties have been encountered in producing tubes from 'Rex 539' steel. One firm has made tubes up to 7-in. diameter and the other reports fabrication of tubing of thicknesses down to 10 s.w.g.

In connexion with the heat-treatment of the steel, experiments have indicated the particular suitability of an electroless nickel-phosphorus deposit for protection against decarburization. Use of an electrodeposited copper coating was found to cause a marked reduction in the elongation values obtained when the steel had been tempered at temperatures below 300°C. Since improvement in elongation as the tempering temperature was raised is associated with release of occluded hydrogen, nickel-phosphorus electroless plating was selected as being less likely to introduce hydrogen and thus induce embrittlement. This assumption was amply justified by the fact that values of elongation on specimens of steel so coated for heat-treatment compare favourably with those obtained on unplated steel treated in a neutral salt bath. In this connexion, it is pointed out that current American practice is to use a flash of nickel-phosphorus plate and follow this with copper electrodeposited from an acid electrolyte. The nickel-phosphorus coating, being amorphous, appears to seal the surface of the steel against ingress of hydrogen during subsequent deposition of copper.

Production of Ultra-High-Tensile Properties in Steels by Deformation and Thermal Transformation

D. J. SCHMATZ and V. F. ZACKAY: 'Mechanical Properties of Deformed Metastable Austenitic Ultra-High-Strength Steel.'

Amer. Soc. Metals, Preprint 110, Oct. 1958; 13 pp.

The increasingly urgent demand for ultra-high-strength constructional materials has led, during recent years, to intensive study of means by which mechanical properties may be improved. The present authors refer to summary reviews of the progress which has been made by use of various techniques, and directs particular attention to the work of LIPS and VAN ZUILEN., who have found that a 33 per cent. increase in yield strength, compared with that obtained by conventional heat-treatments, can be secured by deformation of metastable austenitic alloy steels and subsequent transformation to martensite or bainite (*Metal Progress*, 1954, vol. 66, Aug,, p. 103).

In the study now reported the mechanical properties of steels resulting from deformation (at various temperatures) and subsequent transformation of metastable austenite of varying carbon contents to martensite were determined, and were compared with the properties resulting from conventional heat-treatment of the same steels. The compositions of the steels used are shown in the table below.

The results of the experiments confirm those reported by Lips and van Zuilen. Hardness increase of 2 to 4 points Rockwell C was obtained with deformations ranging from 25 to 75 per cent. and these changes were associated with increase in tensile strength and yield strength. Yield strengths of more than 300,000 p.s.i. (134 t.s.i.; 211 kg./mm.^{2}) were obtained by tempering the steels which had been previously deformed, and at that strength level ductilities of 6–8 per cent. were consistently secured. Electron micrographs of the deformed steels indicate that a refinement of martensite had occurred.

Suggestions are made with regard to the mechanisms responsible for the improvements effected by the procedure described.

Compositions of Ultra-High-Tensile Steels

C %	Si %	S %	P %	Ni %	Cr %
0·98	—	0·004	0·002	3·96	—
0·28	—	0·004	0·004	4·55	1·42
0·35	1·78	0·005	0·001	4·60	1·46
0·40	1·55	0·005	0·004	4·75	1·45
0·49	1·43	0·005	0·002	4·50	1·44

High-Strength Steels for Aeronautical Applications

R. J. NEKERVIS, C. H. LUND and A. M. HALL: 'Status of High-Strength Steels for the Aircraft Industry.' *Battelle Memorial Inst., T.M.L. Report No.* 91, Jan. 3, 1958; 88 pp. + appendices.

The development of supersonic aircraft has confronted the designer with the problem of aerodynamic heating. The suitability of a material of construction for airframe components is governed by its mechanical properties and oxidation-resistance at the service temperature. The use of aluminium alloys is restricted to temperatures below about 400°F. (205°C.); magnesium alloys give satisfactory service at slightly higher temperatures, and those of titanium base are suitable for use up to about 800°–900°F. (425°–480°C.). The limitations of these conventional airframe materials have caused the industry to seek a solution to the problem in steels which offer the advantage of a high strength/weight ratio. Prompted by the increasing interest in the aeronautical applications of the high-strength steels, the Battelle Memorial Institute undertook a survey of actual and potential uses of such materials in the aircraft industry. The results of the survey are given in this report.

The aim of the work was 'to explore the kinds of steel available, the kinds of steel in use, their properties, requirements of aircraft and missile manufacturers, and new developments in steels of potential interest to the aircraft and missile industry'. The emphasis is on sheet steel for airframes of manned aircraft and missiles, but other forms, such as bar stock and forgings, are also discussed. In general, the enquiry was restricted to applications in which the maximum temperature does not exceed about 1200°F. (650°C.). (In this region of temperature the strength of iron-base alloys currently available tends to fall sharply.)

The information on which study is based was obtained from both suppliers and consumers: conferences were held with members of the staffs of twelve steel suppliers and fifteen consumers engaged in manufacturing aircraft and missiles, and information so obtained was supplemented by correspondence, suppliers' data sheets, published literature, and discussions with members of engineering firms.

Six grades of steel were covered by the survey:

(1) *Hot-Work Tool Steels*, exemplified mainly by the chromium-molybdenum-silicon-vanadium steels 'Peerless 56', 'Potomac M' and 'Vasco Jet 1000'.

(2) *Martensitic Stainless Steels*, exemplified by the chromium-molybdenum grades '422', '422M' and '12MoV'.

(3) *Martensitic Low-Alloy Steels*: the chromium-molybdenum-vanadium steel 'Timken 17/22A' is included as typical.

(4) *Chromium-Nickel Precipitation-Hardenable Semi-Austenitic Stainless Steels:* in this connexion reference is made to 'PH15-7Mo', '17-7PH' and 'AM-350'.

(5) *Chromium-Nickel Precipitation-Hardenable Austenitic Stainless Steels:* represented by 'H.N.M.' and 'A-286'.

(6) *Cold-Rolled Austenitic Stainless Steels*, exemplified by the chromium-nickel 18-8 Type 301, the chromium-manganese-nickel Type 201 and 'Tenelon' (a chromium-manganese steel).

The information presented is too diverse and comprehensive to permit of summary, but some indication of its scope can be given, since each of the sections relating to the respective types of steel is similar in format.

In each section, under the heading 'Suppliers' Information', notes are given on composition, heat-treatment, and relevant properties of the particular steel. (Throughout the report, the information discussed in the text is supplemented by comprehensive tabular and/or graphical data.) The advantages and limitations of each steel are critically outlined, and some comparison is made with the other materials reviewed. The second sub-division refers to development work being carried out by various steel manufacturers. The third and final subdivision covers 'Consumer Reactions', a heading which embraces (1) a résumé of work in progress by airframe manufacturers, and applications for which the steel is considered suitable; (2) notes on manufacturing procedures, and the problems which can be anticipated in working or using the material.

In the three other sections which complete the survey, consideration is given, respectively, to the possibility of using the iron-aluminium-molybdenum alloy 'Thermenol' for airframe applications; sizes, tolerances and heat-treatment requirements; data required by the aircraft manufacturer, and the forms in which such information should preferably be presented.

Hydrogen Embrittlement in High-Strength Steel: Influence of Cadmium Plating

J. E. CHILTON: 'Development of Electroplating Processes to Eliminate Hydrogen Embrittlement in High-Strength Steel.' *Wright Air Development Center, Tech. Report* 57-514, Jan. 1958; 77 pp. *P.B.*131721.

KLIER, MUVDI and SACHS, in a comprehensive study of the subject, have shown that the amount of hydrogen introduced into high-strength steels electroplated with cadmium may be as high as that introduced through severe cathodic impregnation (see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 1, pp. 12-13). MORLET, JOHNSON and TROIANO (see *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 49) have sought to interpret the mechanism of embrittlement caused by hydrogen contamination. In an attempt to devise some means of preventing hydrogen embrittlement, BECK and JANOWSKY have investigated the influence, in this respect, of electrodeposition of cadmium and chromium (see *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 43).

The steels selected for study included, in each case, a low-alloy nickel-chromium-molybdenum steel of

the A.I.S.I. 4340 type. The present report relates to a steel of the same grade, heat-treated to tensile strengths in the range 280,000-300,000 p.s.i. (125-134 t.s.i.; 197-211 kg./mm.^{2}). The composition is given as: carbon 0·42, silicon 0·33, manganese 0·7, sulphur 0·008, phosphorus 0·012, nickel 1·92, chromium 0·89, molybdenum 0·22, per cent.

The investigation was carried out in four phases: (1) study of tests used to determine hydrogen embrittlement; (2) study of basis-metal preparation; (3) investigation of sulphamate, cyanide, perchlorate and other cadmium-plating solutions, with the aim of developing an electrolyte which would minimize evolution of hydrogen, exhibit maximum throwing power and give deposits of low porosity and high adhesion; (4) tests to determine the degree of embrittlement caused by plating with the various solutions and procedures used.

From the results of bend, torsion and sustained-load tests, it was concluded that the third test was the most suitable method of evaluating hydrogen embrittlement. In the main test programme static-load fracture tests were therefore carried out, using un-notched specimens.

Of the various methods used to clean the bars prior to plating, de-greasing with trichloroethylene, anodic cleaning in alkaline solutions and vapour, or sand blasting were found to produce no embrittling effects. Cathodic treatment or acid pickling caused severe embrittlement.

In phase (3) commercial cadmium-cyanide solutions were found to have cathode current efficiencies varying from 75 to 85 per cent. (*vis-à-vis* those of 95-100 for the other solutions used). The throwing power of the cyanide electrolyte was much higher than that of the acid solutions, and, when smooth specimens were used, the deposits obtained from the cyanide solution showed highest adhesion. (Satisfactory adhesion was, however, achieved with coatings produced from the acid solutions if the surface of the basis steel had been previously sand blasted.) Corrosion-resistance of deposits obtained from the acid solutions was improved by introduction of addition agents. Hydrogen and cadmium were found to have similar deposition potentials in cyanide electrolytes, whereas, in the sulphamate and perchlorate solutions, deposition of cadmium occurred at a lower potential than did formation of hydrogen.

The results of the static-load fracture tests showed that hydrogen embrittlement was most severe when deposition was carried out from cyanide solutions, somewhat less so when fluoborate electrolytes were used, and least when the cadmium was deposited from sulphamate or perchlorate solutions. Introduction of addition agents to the last three solutions was effective in preventing embrittlement. Addition of formaldehyde and ethylene diamine to cyanide solutions had no value in this respect and the beneficial effect of hydrogen peroxide was only slight. Butyne 1,4 diol and potassium persulphate reduced embrittlement in the case of the acid solutions. Deposition of cadmium-tin coatings, of copper from a pyrophosphate solution, or of a nickel-tin coating from a fluoride solution induced no embrittlement.

Promising approaches for future work are outlined.

Brittle-Fracture Determination by Drop-Weight Test

P. P. PUZAK, A. J. BABECKI and W. S. PELLINI: 'Correlations of Brittle-Fracture Service Failures with Laboratory Notch-Ductility Tests.'

Welding Jnl., 1958, vol. 37, Sept., pp. 391s-407s.

Following serious brittle-fracture failures in welded cargo ships, the Metallurgy Division of the U.S. Naval Research Laboratory has made a systematic study of the mechanical and metallurgical aspects of brittle fracture. An important part of the work has been the development of new tests simulating ultra-sharp notches such as those resulting from the cracking of a brittle weld ('crack-starter weld').

In this report reference is made to some of the earlier stages in the research, and a detailed account is given of notch-bend and drop-weight tests on materials which had developed brittle fracture in service. The failed specimens were representative of the following types of steel: carbon and carbon-silicon ; manganese - molybdenum ; nickel - molyb - denum - vanadium; chromium - molybdenum - vanadium; cast 12% chromium; and low - alloy nickel-chromium.

It is demonstrated that in all cases the NDT (nil-ductility transition) value correlates with the service performance recorded for the respective materials. As predicted by the drop-weight test results, the failures occurred at temperatures below the NDT temperatures established for the various steels. The finding that brittle fractures may start at temperature levels corresponding to the Charpy V-notch 20 ft.-lb. (2·76 kgm.) temperature is of particular significance.

Pack Carburizing and Annealing of Nickel-Chromium-Molybdenum Case-Hardening Steel

C. DAWES: 'The Pack Carburizing and Annealing of 4¼% Ni-Cr-Mo Case-Hardening Steel.'

Metallurgia, 1958, vol. 58, July, pp. 3-9; Aug., pp. 69-75.

Pack carburizing and allied heat-treatment procedures currently used for low-alloy case-hardening steels have been developed mainly on the basis of experience with low-carbon steels. These techniques, however, have not proved entirely suitable when applied to the alloy steels. The carburizing compounds used result, for example, in carbon concentrations of at least 0·09 per cent. in the case, a level which, while advisable with plain carbon steels, is deleterious with the more highly alloyed types, particularly the 4¼ per cent. nickel-chromium-molybdenum steels (*B.S.* En 39). A high carbon content in these steels can (1) produce a massive globular carbide layer in the extreme outer edge of the case which, resulting in serious depletion of the alloyed content of the austenite, raises the M_s-M_f range above that of the inner layer and can cause

cracking during quenching; (2) produce excess carbide in the form of a network of cementite around the grain boundaries, which tends to promote cracking during grinding; (3) cause retention of austenite on cooling from carburizing temperatures, thus complicating annealing characteristics; (4) cause retention of austenite on quenching, giving low hardness values. Carbon concentrations cannot be satisfactorily controlled by varying the carburizing temperature or by using diffusion techniques.

The work described in the first part of this paper was therefore carried out in an attempt to develop a carburizing compound which would achieve the desired effect. Tests were made on two types of steel: 4¼ per cent. nickel-chromium-molybdenum steel and plain carbon steel. Five carburizing compounds, each containing charcoal, but varying in amount and nature of the energizing material, were studied: three were of the impregnated type, in which the water-soluble energizer consisted of 2 per cent. of barium oxide, 1 per cent. of sodium carbonate, or 5 per cent. of sodium carbonate; the fourth was a surface-coated compound using a molasses-bonded energizer of 2½ per cent. barium carbonate; the fifth consisted only of charcoal.

Specimens for metallographic examination were carburized in (1) new compound, and (2) a mixture consisting of 9 parts of old to 1 part of new compound. Carburizing cycles of 2, 6 and 16 hours at 900°C. were used, to give case depths of approximately 0·02, 0·04 and 0·06 in., respectively. The treated specimens were examined by metallographic methods and, in specimens which micro-examination had shown to be promising, carbon and hardness gradients were determined.

The author's conclusions with respect to the nickel-chromium-molybdenum steel are summarized as follows:

'With a 4¼ per cent. nickel-chromium-molybdenum steel, it has been found that a 1 per cent. sodium-carbonate or a 2½ per cent. barium-carbonate compound gives less excess carbide than the more highly energized compounds, and when used as a mixture of 9 parts of old to 1 part of new, both are satisfactory media for carburizing. However, with compounds having low energizer contents, it is essential that the chemical should be adequately bonded to the charcoal to avoid loss under production conditions; this is only ensured by using water-soluble energizers.'

Production factors often require the component to be machined after carburizing: this process is extremely difficult when, as with the high-alloy carburizing steels of the En 39B class, hardness values range from 40 to 60 Rockwell C. The second part of the paper gives particulars of experiments made by the author to determine the shortest annealing cycle capable of producing structures which would render the 4¼ per cent. nickel-chromium-molybdenum case-hardened steel suitable for machining. The percentage of retained austenite in the carburized case is discussed in terms of composition, carburizing temperature and rate of cooling. Consideration of these variables led the author to investigate the influence, on the core and case, of the following factors: critical temperature; cooling rate from the carburizing treatment; sub-critical annealing temperature; multiple tempering; normalizing treatment prior to annealing; isothermal annealing.

Two types of specimen were studied: one having a eutectoid, the other a hypereutectoid, case, of approximately 0·06 in. depth.

The procedures used in these investigations and the results obtained are given in detail. The author's conclusions are quoted below.

'The lower critical temperatures of the case and the core, under normal practical conditions, are shown to be 720°C. and 675°C., respectively.

'Considering the carburized case, it has been shown that the cooling rate after carburizing influences the percentage of austenite retained in hypereutectoid specimens, but it has little effect on eutectoid specimens. Single tempering is effective only on specimens which do not contain retained austenite. Retained austenite does not appear in slowly cooled hypereutectoid specimens, but when it does, i.e., with rapidly cooled specimens, it can be removed by a normalizing or conditioning treatment prior to the sub-critical anneal. Transformation speed during isothermal treatment is slowed down by the presence of retained austenite left from a previous austenitizing treatment.

'With the core, the cooling rate from carburizing has no influence on the ultimate hardness after sub-critical annealing. Multiple tempering softens the core to lower hardness values than single tempering for an equivalent total length of time. Speed of transformation is too slow in the pearlite range for the application of either isothermal or continuous-cooling annealing. This steel can only be annealed, therefore, by the prolonged tempering treatment referred to as sub-critical annealing.

'Summarizing, it can be said that a compromise is necessary in selecting the sub-critical temperature to soften both the case and the core, since if the most suitable temperature below the lower critical is used for the core, transformation is slow in the case.

'With eutectoid cases a useful compromise, employing a multiple tempering technique, is to allow most of the transformation to take place in the case at 700°C.±10°C., cool to atmospheric temperature, and re-heat to 650°C.±10°C., at which temperature transformation of the case is completed and the core is softened.

'With hypereutectoid cases cooled fairly rapidly from carburizing, it is necessary to transform the retained austenite prior to its sub-critical annealing. This is more effectively done by normalizing at 750°C., but this normalize can be omitted if slowly cooled hypereutectoid cases are being considered.'

Production of Chromium-Nickel-Molybdenum-Vanadium-Steel Hollow Propeller Blades

F. J. ALTMANN: 'Fabrication of Hollow Steel Propeller Blades.'

Welding and Metal Fabrication, 1958, vol. 26, Aug., pp. 272–6.

The article gives details of the procedures developed during the war by A. O. Smith Corporation for the production of hollow propeller blades for piston-engine military aircraft.

The chromium-nickel-molybdenum steel used for the blades is covered by *S.A.E./A.M.S. Specifications* 6428 and 6434: percentage limits are given as carbon 0·31–0·37, silicon 0·20–0·35, manganese 0·60–0·85, sulphur 0·025 max., phosphorus 0·025 max., nickel 1·65–2·00, chromium 0·65–0·90, molybdenum 0·30–0·40, vanadium 0·17–0·23. The vanadium addition permits the use of higher tempering temperatures without decrease in tensile strength: a higher degree of stress relief is thus attained, ductility is improved and notch-sensitivity is lessened.

The blade is fabricated from sixteen components, production of which involves precision forging and contour rolling. The parts are then assembled by flash welding and the composite is heat-treated by die-quenching and tempering. The blades are straight and flat before quenching, and the final twist and generation of the correct airfoil section is effected simultaneously with the die-quenching operation. Finally, the root is machined, the blade is carefully balanced, and the bearing mechanism is assembled.

The salient features of each of these processes are outlined by the author. The same basic techniques have been extended to production of such items as airframe and jet-engine components, missile bodies, and steam-turbine blades. Stainless steel, heat-resisting alloys and titanium alloys have also been processed by such methods.

Welding of 3½% Nickel-Chromium-Molybdenum Steel

C. L. M. COTTRELL and K. WINTERTON: 'Weldability of 3½% Ni-Cr-Mo Steel.'

Brit. Welding Jnl., 1958, vol. 5, Oct., pp. 481–4.

It had been shown by earlier work that the tendency of alloy steels to cold cracking in the heat-affected zone during metal-arc welding is related to the temperature of completion of the austenite transformation during cooling at the given welding rate. This temperature was found to depend upon the type of welding electrode used, and to be lowest for electrodes which gave welds of low diffusible-hydrogen content. Critical end-of-transformation temperatures of 290° and 245°C. were established for rutile-coated ferritic and low-hydrogen ferritic electrodes, respectively. It was also shown that if there was a critical temperature for rutile-coated austenitic electrodes it was below about 205°C.

Parallel investigations had shown that cold cracking could be simulated in a dead-load tensile test piece. A delayed brittle fracture was produced at low stress levels when conditions (hydrogen content and cooling rate) which produced cracks in commercial practice were simulated in the test piece immediately before application of the dead load. (The *bibliography* attached to this report gives references to the original papers in which these results were published: see, for example, abstracts in *Nickel Bulletin*, 1953, vol. 26, No. 7, p. 116; 1954, vol. 27, No. 10, p. 195.)

The work has been continued on a 0·3% carbon, 3½% nickel-chromium-molybdenum steel (*B.S.* En 28) used in the form of 0·8-in. and 1-in. thick plate in the hardened-and-tempered condition. The metal-arc welding electrodes used were of 18–8 chromium-nickel steel: the ferritic welding wire was of the silicon-manganese steel type.

The programme of investigation included (1) CTS weld tests, (2) rapid dilatation tests on tubular specimens heated to peak temperatures of 1240° or 1300°C. and cooled at six different rates, and (3) slow tensile and dead-load tensile tests on notched specimens of the steel after they had been heated to a peak temperature of about 1300°C. and cooled at three rates. Some specimens were heat-treated after degassing by vacuum heating up to 950°C.: they contained 0·35 ml./100 g. less hydrogen than the standard specimens.

The results of the tests indicate that

Since considerable cracking was observed in welding with a cooling time (870°–300°C.) of about 20 seconds, end-of-transformation temperature must be considerably higher than 70°C. to give crack-free welds when using austenitic electrodes (almost zero diffusible hydrogen). Critical end-of-transformation temperature for steels welded with austenitic electrodes is thus likely to be between 70° and 200°C. and the same range of temperature may also apply for steels welded with bare ferritic wire (argon-shielded). There is some suggestion that, contrary to general belief, the M_S temperature of this steel may be affected by the rate of cooling.

'Mechanical tests on simulated weld-heat-affected zones have shown that residual hydrogen in the steel (0·35 ml./100 g.) is sufficient to lower the dead-load rupture strength to a value below 40 tons per sq. in. (63 kg./mm.²) after simulation of the thermal cycle involved in welding. The better results obtained on the degassed samples gave strong support to this view.'

Materials for Aerodynamic Applications

See abstract on p. 340.

Welding of Dissimilar Metals: Technique, Electrodes and Welding Wire

See abstracts on p. 350.

Determination of Tungsten in Steels

BRIT. IRON AND STEEL RESEARCH ASSOCN., METHODS OF ANALYSIS COMMITTEE: 'The Determination of Tungsten in Steel not containing Niobium and Tantalum.'

Jnl. Iron and Steel Inst., 1958, vol. 190, Sept., pp. 51–5.

The procedure laid down in *B.S.* 1121: *Part* 3: 1948: for determination of tungsten in steels is based on reduction of iron to the ferrous state and hydrolysis precipitation of tungstic acid with the aid of rhodamine B. In the case of molybdenum-containing steels, co-precipitation of molybdic oxide with the tungsten-hydrolysis product has tended to give results which are too high. The procedures were therefore referred, for examination, to the B.I.S.R.A. Methods of Analysis Committee: the results of their investigations are reported in this paper. The range of materials examined was restricted to steels free from niobium and tantalum.

After investigation of various other techniques, which proved unsuitable, a study was made of determination of tungsten as tungstic oxide, using sulphurous-acid hydrolysis with cinchonine as auxiliary precipitant. Results for high-alloy molybdenum-tungsten steels were satisfactory; those for low-tungsten steels were erratic. Determination of tungsten in steels containing up to 3 per cent. of the element was, however, already provided for by the photometric thiocyanate procedure described in *B.S.* 1121: *Part* 32. Further work was therefore concentrated on application of the gravimetric method to determination of tungsten in the range 3–32 per cent. Full details are given of the procedure finally evolved. The technique has been submitted to the British Standards Institution with a view to its superseding that recommended in *B.S.* 1121: *Part* 3, issued in 1948.

HEAT- AND CORROSION-RESISTING MATERIALS

Research at the Birmingham Laboratory of The Mond Nickel Company, Ltd.

'Development of High-Temperature and Other Special Alloys: Mond Nickel Research Laboratory.'

Metal Industry, 1958, vol. 93, Oct. 24, pp. 355–8.

The Development and Research Department of The Mond Nickel Company, Ltd. operates two research laboratories: one at Birmingham the other at Acton. Work at the Birmingham Laboratory relates primarily to nickel and nickel-containing materials; the Acton Laboratory is concerned with research on the platinum metals. The staff of 220 at the Birmingham Laboratory is divided into 13 teams, each of which deals with a specific field of metallurgy (e.g., steel, cast iron, high-temperature materials, corrosion, plating, welding, analysis, mechanical testing). In this article the wide range of activities covered by these research teams is illustrated by an outline of some of the more important work currently in progress.

In connexion with research on steels, reference is made to work, on high-purity steels, which illustrates the relationship between composition and embrittlement. Mention is made of the Company's research programme relating to spheroidal-graphite and high-alloy cast irons, and notes are given on the 'Nimonic' alloys which, developed in the Birmingham Laboratory specifically for rotor blading of British aircraft gas turbines, have remained the standard material for this application over a period of 17 years. Work on corrosion-resisting materials is exemplified by reference to 'Corronel B', a nickel-molybdenum-base alloy resistant to concentrated hydrochloric acid and to a nickel-chromium alloy which is highly resistant to concentrated nitric acid. The Physics Section of the Laboratory is concerned mainly with the study of nickel-containing alloys having closely controlled magnetic or electrical properties: the scope of current investigations is indicated. Finally, reference is made to work being carried out in relation to powder metallurgy and welding.

Hereford Works of Henry Wiggin and Company, Ltd.

See abstract on p. 320.

Nickel-base Alloys and Stainless Steel: Literature Reviews

Industrial and Engineering Chemistry of September 1958, vol. 50, No. 9, Part II, contains The Eleventh Annual Review of Unit Processes in Chemical Engineering and The Eleventh Annual Review of Materials of Construction.

As in earlier reviews, the articles are based on the literature which has appeared since the previous surveys. Two items in the section on materials of construction are of specific interest in relation to nickel-containing alloys and steels. Their scope is indicated below.

A. J. MARRON: 'Nickel, including High-Nickel Alloys', pp. 1460–9.

The review (based on a *bibliography* of 181 items) is divided into four main sections, covering, respectively: physical metallurgy, research and development; materials for high-temperature applications; other applications of nickel and nickel-base alloys; welding and fabrication.

Features of the literature to which reference is made include the following:

Exploitation of new nickel deposits by The International Nickel Company, Inc., which has ensured ample and dependable supplies of nickel, sufficient not only to meet the needs of all present users, but also to expand the range of applications of the metal to many fields of industry which have hitherto been deprived of its use.

Research on the physical, thermal, electrical and magnetic properties of nickel and its alloys, corrosion- and oxidation-resistance, sorption and diffusion.

Applications of high-nickel alloys in the petroleum, nuclear energy and other industries; aeronautical,

missile, turbine and electronic applications; investigation of properties relevant to these applications.

Advances recently made in welding and brazing techniques, including development of welding electrodes suitable for nickel alloys and clad materials.

W. A. LUCE and J. H. PEACOCK: 'Stainless Steels and other Ferrous Alloys', pp. 1482–8.

Considerable attention continues to be given to the various technological aspects of the austenitic stainless steels. Stress corrosion of these materials is becoming a major problem and research into the phenomenon is being intensified. Reference is made to work on the resistance of the stainless steels to various corrosive media: that on resistance to attack by high-purity water reflects the growing importance of these materials in nuclear energy applications. Literature cited in relation to mechanical properties includes reports on behaviour at sub-zero temperatures and on the effects of strain- and age-hardening. With respect to physical metallurgy, papers have been published on structural changes at high temperature, and particular interest has centred on sigma formation and transformation of delta ferrite. A résumé is also given of investigations relating to the properties of precipitation-hardenable and other heat-resisting stainless steels. The review of welding developments contains allusions not only to investigations of the general welding characteristics of the austenitic steels, but also to procedures which have been proved suitable for specific applications. In the section covering general aspects of recent development work, results of investigations relating to modified steel compositions are summarized, and advances in manufacturing and processing techniques are noted.

The *bibliography* comprises 137 items.

Vacuum Melting of High-Temperature Alloys

The three articles to which attention is directed below relate to the production of materials by vacuum-melting techniques. The first two are essentially review articles: the first outlines the processes and procedures used, the second refers to factors requiring consideration if contamination is to be minimized. The information contained is specific to no one alloy, but is presented in terms of its general applicability to the 'superalloys' (i.e., those of nickel-base containing such chemically active elements as titanium, aluminium, zirconium, or boron) for which vacuum melting has proved peculiarly suitable. The third article forms an interesting supplement to the others, in that it describes the problems involved in, and procedures evolved for, production of a specific high-temperature alloy by vacuum melting.

'Vacuum Melting Today.'

Metal Progress, 1958, vol. 74, Aug., pp. 94–9.

The advantages and limitations of vacuum induction melting and consumable-electrode re-melting are discussed, and a brief description is given of the procedures involved. The article also contains notes on de-gassing of melts by pouring *in vacuo*, quality control, forging and rolling of vacuum-melted ingots, inspection, and the principal applications of vacuum-melted alloys. Compositions of vacuum-melted steels and alloys available in the U.S.A. are listed.

D. E. NULK: 'A User Looks at Cleanliness of Vacuum-Melted Alloys.'

Ibid., pp. 103–9.

'Cleanliness' in vacuum-melted alloys (the emphasis in the article is on those used for gas-turbine components) is discussed in the light of the deleterious effects of contaminants and methods of preventing their occurrence.

By way of introduction, the author refers to types of contamination which have proved particularly troublesome in vacuum melting. In this connexion, size, shape and distribution of carbides, the presence of non-metallic inclusions, and the necessity of controlling grain-size are discussed. The effects of boron in high-temperature alloys are summarized, with an indication of the deleterious effects of formation of compounds with nitrogen and oxygen: when combined with critical alloying elements (e.g., titanium or boron) such contaminants could render their presence ineffective. The relationship between homogeneity and ease of fabrication is considered, and reference is made to the adverse effects of stringers on the service life of the alloy.

Finally, a brief outline is given of improvement in vacuum systems and in melting practice which have contributed to production of cleaner alloys, and current methods of evaluating cleanliness are described (the modified A.S.T.M. inclusion rating, frequency/severity ratings and metallographic classification).

D. R. CARNAHAN: 'Production of 'Discaloy' by Vacuum Arc Melting.'

Ibid., pp. 100–2.

The article gives details of the work involved in developing a satisfactory procedure for consumable-arc re-melting of 1200-lb. (540-kg.) ingots of 'Discaloy' (the composition of which is given on p. 332). Production was on a pilot scale and use was made of a vacuum-arc furnace which could be operated at pressures of 2–10 microns.

The main difficulties were encountered in the preparation of the electrodes. In the as-cast condition they were found to be unsuitable, producing excessive gas during melting; moreover, any contamination in the electrodes was reproduced in the vacuum-melted ingot. These problems were overcome by use of electrodes fabricated from 7-in. (17-cm.) round billets machined from ingots which were were known to be internally sound. Preliminary investigations showed that the starting pads, on which the arc is first struck, should be cut from vacuum-arc-melted 'Discaloy' ingots.

Determination of optimum melting rates was made in the light of the need for uniform solidification and a sound surface in the ingot. Pipe cavities were minimized by reducing the power input during, and increasing the time of, hot-topping. Use of a magnetic stirring coil was discontinued when it was found to result in segregation.

The melting procedure finally evolved is summarized as follows:

'The electrodes used were 17-ft. (5-m.) long; to fabricate them we welded two 7-in. (17-cm.) diameter machined 'Discaloy' billets. All billets were thoroughly tested for internal soundness. With a starting pad of the same grade (which had been consumable-arc melted), melting began. After an initial rise in pressure, the vacuum was maintained at 4 microns, under a steady current of 7000 amp. Using 22-24 volts, melting took about three hours for each ingot, including the 20-minute hot-topping schedule.'

Comparative tests on vacuum- and air-melted ingots showed that in the former segregation was much less and that mechanical properties were significantly higher.

'W-545': Iron-Nickel-Chromium-base Alloy

J. T. BROWN: ''W-545' . . . A Better Turbine Disk Alloy.'

Metal Progress, 1958, vol. 74, Aug., pp. 87–90.

The article gives details of the development, by Westinghouse Electric Corporation, of a precipitation-hardenable alloy designated 'W-545'. The composition of the alloy is based on that of the age-hardenable alloy 'Discaloy': see table below.

Addition of 0·005–0·15 per cent. of boron to 'Discaloy' was found greatly to increase ductility, without affecting time-to-rupture. This increase in ductility enabled the percentage of hardening agent (previously restricted to about 2 per cent. of titanium) to be increased to 3·5 per cent., the limit of solid solubility, while still retaining a satisfactory degree of ductility. The resultant material is therefore much stronger and has a longer creep-rupture life under a given stress.

Stress-rupture tests showed that with a titanium content of about 1·7 per cent. addition of 0·005 per cent. boron was sufficient to achieve satisfactory ductility, and that a boron content greater than 0·007 per cent. did not affect the properties obtained. Elongation and time-to-rupture are plotted, in a three-dimensional graph, as a function of boron content: elongation at rupture is found to decrease with increase in rupture time and to increase with increase in boron content.

The influence of boron content on the notch sensitivity of the alloy was studied by stress-rupture tests at 1200°F. (650°C.) under 70,000 p.s.i. (31 t.s.i.; 49 kg./mm.²), on notched and un-notched specimens. Boron content was varied in the range 0–0·12 per cent. The influence of heat-treatment on stress-rupture properties was established by subjecting the specimens to widely varying heat-treatment cycles: the results are tabulated. Increase in boron content improved the ductility of the un-notched specimens: at an elongation of approximately 5 per cent. rupture times for notched specimens became longer than those for the un-notched test pieces. Variation in heat-treatment was found significantly to affect the properties obtained: size and distribution of the precipitate phase are believed to govern the strength and ductility of the alloy.

The remainder of the paper relates to mechanisms postulated in explanation of the effect of boron.

Production of the alloy has reached the stage of commercial development, and typical mechanical properties and stress-rupture data obtained from wrought products made from commercial-size ingots are reported.

Cast Age-Hardenable Nickel-Chromium Steels

E. A. LANGE, N. C. HOWELLS and A. BUKOWSKI: 'Cast Age-Hardenable Austenitic Steels.'

U.S. Naval Research Laboratory, Report 5140, May 5, 1958; 14 pp.

Naval engineers designing hardware for mine sweepers are handicapped due to lack of suitable high-strength non-magnetic casting alloys. This report describes work carried out to study the possibility of adapting three types of wrought age-hardenable austenitic steels for use as castings. A fourth type of steel which has no exact counterpart was also investigated, because it had particular interest due to its low alloy content. The composition ranges of the materials studied are shown in the table on page 333.

Detailed investigation of the influence of variation in composition and treatment, on the properties and structure of the respective steels, led to the following conclusions:

Chromium–Nickel–Phosphorus Steels

This type of steel can be cast, solution-treated and age-hardened to attain yield strengths of more than 100,000 p.s.i. (44·5 t.s.i.; 70·5 kg./mm.²). The composition giving optimum tensile properties was

	Ni %	Cr %	Mo %	Ti %	B %	Fe %
'Discaloy'	26	13	3·0	1·8	—	Bal.
'W-545'	26	13	1·5	2·8	0·03	Bal.

Chemical Composition of Four Types of Cast Age-Hardening Austenitic Steel

Type	Composition Range						
	Cr %	Ni %	Mn %	C %	P %	V %	Mo %
1. Cr-Ni-P	16·5-19·0	8·0-11·0	0-3·5	0·1-0·4	0·1-0·3	—	—
2. Cr-Mn	12·0-16·0	—	12·0-16·0	0·1-1·0	0-0·3	0-1·2	—
3. Cr-Mn-Ni	4·5-6·0	5·5-9·0	9·0-11·0	0·5	—	0·3-1·1	—
4. Mn-V	0-4·5	2·0-4·0	14·0-18·0	0·25-1·2	—	0·25-1·2	0·4-3·5

chromium 18, nickel 10, manganese 3·5, carbon 0·3, phosphorus 0·25, per cent.

Maximum hardness was attained by the following heat-treatment: solution annealing for one hour at 2100°F. (1150°C.), followed by age-hardening for 8 hours at 1350°F. (730°C.) or for 16 hours at 1300°F. (705°C.).

Ductility (% elongation) ranged between one and four per cent. Further research on heat-treatment may be expected to improve ductility in the aged condition, while maintaining a high level of strength.

It is necessary, in production, to minimize the hydrogen content of the metal, prior to and during casting, in order to avoid excessive segregation and possible exudation of the phosphorus constituent during solidification and the solution heat-treatment.

Chromium-Manganese Steels

Addition of 0·25 per cent. phosphorus, with 0·3 per cent. carbon, promotes age-hardening characteristics in this steel, but the chromium-manganese-phosphorus steels are more prone to cracking than chromium-nickel-phosphorus types.

Age-hardening can be induced in chromium-manganese steels containing vanadium. No specific composition can be recommended for castings in this type of steel, but those containing more than 13 per cent. manganese and 2 per cent. nickel are recommended for further research.

Chromium-Manganese-Nickel-Vanadium Steels

The carbon and vanadium contents usual in wrought steels of this type must be lowered for castings, in order to obtain a satisfactory level of ductility. The composition recommended, to maintain correct balance between strength and ductility in the age-hardened condition, is chromium 5·7, nickel 8·0, manganese 7, vanadium 0·4, molybdenum 3·3, carbon 0·5, per cent. The authors do not recommend any further development work on this group of steels, since austenitic steels of lower alloy content have comparable strength levels.

Manganese-Vanadium Steels

Age-hardening characteristics can be induced in these steels: a minimum of 0·3 per cent. vanadium is necessary in order to obtain yield strengths of 100,000 p.s.i. (44·5 t.s.i.; 70·5 kg./mm.²). Molybdenum intensifies vanadium in the age-hardening reaction, but a minimum of 0·2 per cent. vanadium must be present. Chromium should be less than 2 per cent.

'Light-Zone' Phenomenon in Age-Hardened Nickel-Chromium and Nickel-Chromium-Cobalt Alloys

C. BÜCKLE and J. POULIGNIER: 'Identification and Study of 'Light-Zone' Phenomenon in Nickel-Chromium and Nickel-Chromium-Cobalt Age-Hardenable Alloys.'

Rev. Métallurgie, 1958, vol. 55, May, pp. 417-29; disc., pp. 429-32.

The study described was carried out in an attempt to gain more information on the mechanism and effects of the zig-zag light-coloured regions which have been variously reported to occur along the grain boundaries of nickel-chromium and nickel-chromium-cobalt high-temperature alloys during ageing. (The grain-boundary zones are similar to the 'light-zone phenomenon' observed in aluminium-base alloys.)

Commercial alloys were selected for investigation. Numerous micrographs are presented in support of the authors' findings and in illustration of the metallographic techniques by which the study was carried out.

The paper is presented in four sections. After outlining the significance of rate of cooling after solution treatment, the authors describe methods used to identify the crystallographic features of the structure and discuss the conclusions they have reached. Metallographic methods of determining the evolution of the zone as a function of time and temperature of treatment are considered, and finally an attempt is made to determine the influence of the structure on creep-resistance.

To determine the effect, on the occurrence of the phenomenon, of rate of cooling from the solution-annealing temperature, specimens of both alloys were water-quenched or air-cooled, subjected to the usual ageing treatment, and examined metallographically. The water-quenched specimens were found to contain the zig-zag grain-boundary zones; those which had been air-cooled showed no sign of the phenomenon. Slight variations in alloying content did not influence these results.

The crystallographic features of the zone may be determined in two ways: by colour-etching, and by observation of slip lines formed by microhardness indents. The results of microexamination by these methods are presented in confirmation of the theory that the zones result from localized recrystallization at the grain boundary: this is caused by interpenetration of neighbouring grains, and is accompanied by diffusion and irregular precipitation.

The evolution of the zone was also studied in two ways: by examination of the specimens after ageing times of 30 minutes and 311 hours; by interrupting the ageing treatment at short intervals and so determining the progress of the zone after, for example, 2, 5 and 15 minutes. The micrographs show that the formation of the zones is time-dependent, but that the ageing temperatures used (700°C. for the nickel-chromium and 750°C. for the nickel-chromium-cobalt alloys) have no influence in this respect.

Creep tests were carried out on specimens water-quenched or air-cooled from the solution-treatment temperature. Although, in general, the creep life of quenched specimens was lower than that of those air-cooled, and the creep rate was higher, it is concluded that there is no evidence that these variations were connected with the zones being examined. Metallographic examination appeared to indicate that during creep the material forming these zones is endowed with a certain plasticity which might even prove beneficial.

Influence of Heat-Treatment on Microstructure and Properties of a Complex Nickel-Chromium-Cobalt-base Alloy

R. F. DECKER, J. P. ROWE, W. C. BIGELOW and J. W. FREEMAN: 'Influence of Heat-Treatment on Microstructure and High-Temperature Properties of a Nickel-base Precipitation-Hardening Alloy.'

Nat. Advisory Committee for Aeronautics, Tech. Note 4329, July 1958; 53 pp.

It is now generally accepted that the favourable high-temperature properties of nickel-base alloys hardened by titanium and aluminium result from precipitation of the intermetallic γ' phase within the matrix of the alloy. Some of the literature relevant to the influence of the γ' phase is reviewed in the introduction to this report: the discussion relates mainly to alloys of the 70–80 per cent. nickel type, e.g., the 'Nimonic' alloys and 'Inconel X'.

The experimental work reported formed part of a broad programme carried out at the University of Michigan to study the basic mechanisms by which processing variables influence the high-temperature properties of nickel-base alloys hardened with titanium and aluminium. A specific aspect considered in selecting the composition for study was extension of knowledge to nickel-base alloys containing the higher amounts of titanium and aluminium currently being used. The full composition of the alloy tested is shown in the table below.

The work comprised investigation of the effects, on (1) size and distribution of precipitates, and (2) high-temperature properties, of the following conditions:

Solution-treatment temperature.
Rate of cooling after solution treatment.
Isothermal ageing after solution treatment.

Assessment of the effects of these variables was made by hardness measurements, X-ray-diffraction studies, and stress-rupture testing at 1600°F. (870°C.) under a stress of 25,000 p.s.i. (11·15 t.s.i.; 17·5 kg./mm.^{2}), using 0·250-in. diameter specimens machined from heat-treated bar stock.

From the very large amount of experimental data recorded, the following main conclusions were derived:

The most striking microstructural characteristic of this type of alloy is the large amount of the intermetallic γ' phase which precipitates within the matrix grains, and the rapidity with which it forms. The temperature required for complete solution of precipitates was found to lie between 1975° and 2000°F. (1080° and 1093°C.) and the temperature at which the alloy was solution-treated exerted a marked effect on stress-rupture properties. Solution treatment at 1975°F. (1080°C.) resulted in higher rupture life than that obtained by treatment at 2150°F. (1176°C.), or by successive treatment at 2150°F. (1176°C.), and at 1975°F. (1080°C.). The experimental evidence indicates that this difference was due not to lack of complete solution of the γ' phase, but to the retention, in the alloy treated only at 1975°F. (1080°C.), of residual effects from prior rolling operations.

The rate of cooling after solution treatment also affected stress-rupture properties: very rapid or extremely slow cooling (ice-brine quenching or furnace cooling) resulted in rupture properties inferior to those attained by moderate cooling, e.g., by air. These effects were related to the distribution of the γ' phase: furnace cooling caused over-ageing, with coarse dispersion of agglomeration of γ' particles; ice-brine quenching induced considerable cellular precipitation of the γ' phase at the grain boundaries.

Double-ageing treatments after solution had no significant effect on stress-rupture properties. The investigation included also qualitative comparisons of the effects of isothermal ageing, with and without application of stress.

Chemical Composition of Complex Nickel-Chromium-Cobalt-Alloy

Cr %	Co %	Mo %	Ti %	Al %	Mn %	Si %	Fe %	Zr %	C %	B %	S %	Ni %
18·8	15·1	4·15	3·14	3·14	<0·10	0·10	<0·30	0·19	0·08	0·0005	0·008	Balance

The general conclusion is that stress-rupture properties of this alloy can be related to the size and dispersion of the particles, provided that residual rolling effects are removed by the solution treatment. All heat-treatments giving finely dispersed γ' particles throughout the microstructure gave essentially equal properties at 1600°F. (870°C.) and 25,000 p.s.i. (11·15 t.s.i.; 17·5 kg./mm.²). Treatments causing over-ageing in the matrix or at the grain boundaries before testing resulted in significantly inferior high-temperature properties.

Diffusion in the Aluminium-Nickel System

See abstract on p. 321.

Strengthening of High-Temperature Alloys by Admixture of Refractory Oxides

J. D. BURNEY: 'A Study of the Possibility of Reinforcing High-Temperature Alloys by Addition of Refractory Powders.'

Wright Air Development Center, Tech. Report 150971, Feb. 1958; 42 pp.

The report is one of a series covering a general investigation of the possibility of improving high-temperature strength of 80–20 nickel-chromium alloys by admixture of refractory powders.

In earlier stages of the research the examination of composites had shown that adequate dispersion of the powder constituents had not been obtained, and in view of the likelihood that in the SAP process severe working of the metal/refractory-powder material is beneficial in this respect, further experiments were carried out to determine whether such treatment would aid in production of nickel-chromium/oxide composites. The oxides used (separately) were TiO_2 and Al_2O_3, both in ultra-fine form.

The fabricating methods used were: (1) pressing and sintering; (2) pressing and sintering followed by hot- and cold-working treatments; (3) liquid-phase sintering; (4) internal-oxidation techniques.

Preliminary assessment of the quality of the cermets made by the various methods was on the basis of room-temperature impact tests and short-time elevated-temperature stress-rupture tests. Materials which, under such tests, showed promise were further evaluated by

(1) Room-temperature tensile testing.

(2) Impact testing at 1800°F. (982°C.).

(3) Thermal-shock testing at 2000°F. (1093°C.), using water as the quenching medium.

(4) Oxidation testing at 2000°F. (1093°C.) in still air.

(5) 100-hour life stress-rupture testing at 1800° and 1975°F. (982° and 1080°C.).

The structure of the cermets was correlated with their mechanical properties, and throughout the report comparison is made of the properties of these materials with those of wrought 80–20 nickel-chromium alloys. In view of the successful results obtained by the liquid-phase sintering method, in which carbon was an integral constituent of the mass, some tests were made also on an 80–20 nickel-chromium alloy into which carbon was introduced as lamp black.

Results of the individual series of tests are reported *in extenso*. The major conclusions drawn from a general survey of the results are as follows:

Using the press-sinter method of production, the best stress-rupture properties at 1600°F. (870°C.) were obtained with additions of 3·0–4·5 per cent. of TiO_2. The 4·5 per cent. addition gave a rupture time of 29·6 hours, as compared with 1·0 hour on bars containing no TiO_2. The various hot- and cold-working treatments applied gave no improvement in stress-rupture properties.

The liquid-phase sintering technique yielded the most successful results. Stress-rupture tests at 1800°F. (982°C.) showed a stress of 6,200 p.s.i. ($2\frac{3}{4}$ t.s.i.; 4·35 kg./mm.²) for an 80–20 nickel-chromium alloy containing 1% TiO_2 + carbonaceous material. This value was threefold that of the 80–20 nickel-chromium alloy under identical conditions. Tests on the same materials at 1975°F. (1080°C.) showed that the liquid-phase-sintered material had twice the strength of the non-oxide-containing alloy. Room-temperature strengths of the metal/oxide composites were consistently of the order of 75,000 p.s.i. (33·5 t.s.i.; 52·75 kg./mm.²). The thermal-shock properties of the metal/oxide materials were adequate, as evidenced by the fact that they withstood 25 thermal-shock cycles (with water as the quenching medium) without signs of cracking. The oxidation-resistance of the composites was considered good, although not quite so high as that of 80–20 chromium-nickel alloy.

Creep Testing by the Cantilever Bending Method

G. T. HARRIS, H. C. CHILD, A. B. COLLIER and C. F. WEST: 'Recent Developments in Creep Testing by the Cantilever Bending Method.'

Jnl. Iron and Steel Inst., 1958, vol. 190, Oct., pp. 136–43.

When the creep-testing unit described in this paper was first developed (see HARRIS and CHILD, *ibid.*, 1950, vol. 165, June, pp. 139–44) the cantilever bending method was selected mainly because it permitted the use of a very simple specimen which could be precision-cast or produced by powder-metallurgical techniques. Experience soon showed, however, that this method could be used for a much wider range of applications than the relatively crude screening tests for which it was originally intended. It therefore became necessary to improve the design and sensitivity of the test apparatus. The design of the units now in use, and the type of work for which they are employed, are reported in this paper. Results obtained with the cantilever method are correlated with tensile data.

The creep-testing units described are capable of carrying out tests at temperatures up to 1200°C., at stresses of 50 p.s.i. – 50 t.s.i. (0·035–79 kg./mm.²) with a sensitivity of strain measurement of $1\text{–}3 \times 10^{-6}$.

Three machines are described: (1) for use at temperatures up to, and (2) above, 950°C., and (3) for testing *in vacuo*. Specimen dimensions are given for each type, and interpretation of the results is discussed.

The uses of the units are exemplified by a description of tests selected as typical of the following applications: alloy development where the stress for low creep strains is the criterion; evaluation of microspecimens; creep tests on precision-cast alloys; tests on unmachinable materials (e.g., titanium carbide-base materials); creep tests above 1000°C. (overcoming problems of extensometry and clamping); creep tests on sheet specimens; creep tests *in vacuo* or in protective atmospheres. The data presented relate to the nickel-chromium-base alloys 'W7', 'G 39' and 'Nimonic 80', the cobalt-chromium-nickel-base alloys 'G 42B' and 'G 32', and the chromium-nickel-cobalt steel 'G 19'.

It is stated that where a comparison has been made between cantilever and tensile results, agreement has generally been within ±10 per cent.

Influence of Creep on the Short-Time Mechanical Properties of '17-7 P.H.' Steel

J. V. GLUCK, H. R. VOORHEES and J. W. FREEMAN: 'Effects of Prior Creep on Mechanical Properties of Aircraft Structural Metals.'

Wright Air Development Center, Tech. Report 57-150, *Parts I* and *II*. *P.B.*131716.

When alloys and steels are subjected to operational conditions involving stress at high temperatures, account must be taken not only of the possibility of structural changes which might adversely affect the mechanical properties of the material, but also of the effects of creep (i.e., the likelihood of its accelerating the temperature-induced changes, as well as its mechanical effects *per se*). Such conditions obtain also for short durations in the structures of high-speed aircraft, and ability to withstand their effects without significant alteration in short-time mechanical strength, ductility and shock-resistance is a prerequisite of the material of construction. Little information is, however, available on the effects of creep in this connexion. The work described in the two sections of the report referred to below comprises part of a programme to obtain such data for three aircraft structural materials, in sheet form: an aluminium alloy, a titanium alloy, and the chromium-nickel steel '17-7 P.H.'. Test conditions were selected to simulate those obtaining in aircraft structures subjected to aerodynamic heating.

Part I, Feb. 1958; 106 pp.

This part of the report relates mainly to the results obtained for the aluminium alloy. Work on the '17-7 P.H.' stainless steel was limited to a survey of the effects, on room-temperature tensile properties, of prior exposure, at temperatures in the range 600°-900°F. (315°-480°C.), under stresses selected to give a total deformation of 2 per cent. Specimens were exposed in this range at temperature intervals of 50F°. (28C°.) in order to establish the temperature at which exposure to creep had the maximum effect. (In the investigations described in Parts I and II of the report the data obtained relate to the steel in the TH-1050 condition: for details of this condition see *Nickel Bulletin*, 1958, vol. 31. No. 2, pp. 52-3.)

The results obtained showed that

(1) Exposure, at all the test temperatures, with or without stress, tended to raise the tensile and yield strengths.

(2) The effect of exposure on stressed specimens was greater than that found for unstressed specimens, particularly in reducing the range between the tensile and yield strengths.

(3) The maximum effect of exposure occurred within the range 800°-850°F. (425°-455°C.).

(4) Exposure under stress within the lower range of the test temperatures (600°-750°F.; 315°-400°C.) was found greatly to reduce ductility at room temperature.

Part II, Apr. 1958; 91 pp.

Part II of the report is concerned entirely with the data obtained from a comprehensive series of tests on the '17-7 P.H.' steel.

Specimens of the steel were exposed, at 600°, 800° and 900°F. (315°, 425° and 480°C.) for 10, 50, or 100 hours, to stresses selected to achieve a total deformation of 0·5, 1, 2, or 3, per cent. during the exposure period. Other specimens were subjected to the same temperatures for the same times, but with no applied stress. To determine the effects of the various test conditions, on the mechanical properties of the steel, the following series of tests were carried out on each specimen after exposure : tensile and compressive tests at room- and exposure temperature; tension-impact tests (on notched and un-notched specimens) at room- and exposure temperature: determination of hardness at room temperature.

In general, exposure of the steel to the test conditions resulted in remarkably little change in short-time mechanical properties. The major changes determined were in the ductility and compressive yield strength of specimens subjected to creep at 600°F. (315°C.): values for these properties were found to be lowered when subsequently tested at 600°F. and room temperature. Changes observed in other properties, either at room temperature or at the temperature of exposure, were relatively small, and were found, in most cases, to be hardly outside the scatter exhibited by the data.

Such changes in properties as did occur as a result of exposure to creep at 800° and 900°F. (425° and 480°C.) were generally beneficial, and there was no indication of any significant decrease in any of the mechanical properties studied.

Creep appeared to be an essential factor in lowering ductility and compressive yield strength during exposure at 600°F.: little change in these properties was observed in unstressed steel or in specimens

which exhibited only a small amount of creep. The limited metallographic examination which was undertaken revealed that exposure to stress and temperature had had little, if any, effect on microstructure.

Compressive Strength and Creep Properties of '17-7 P.H.' Steel Plate

B. A. STEIN: 'Compressive Strength and Creep of '17-7 P.H.' Stainless - Steel Plates at Elevated Temperatures.'

Nat. Advisory Committee for Aeronautics, Tech. Note 4296, July 1958; 33 pp.

Precipitation-hardening stainless steels are finding increasing use in aircraft structures, due to the fact that in the annealed condition they can be easily fabricated and that the desired strength properties can be subsequently developed by heat-treatment. Since plates are important load-carrying members of aircraft structures, the investigation reported was undertaken to obtain experimental data on a typical precipitation-hardening steel which could be used in such applications. The work was supplementary to an investigation on aluminium-alloy plates.

Compressive-strength tests were made from room temperature to 1000°F. (540°C.) and compressive-creep-test results are given for the range 700°-1000°F. (370°-540°C.). The plate was tested in the TH-1050 condition (for explanation of this terminology, see *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 52). The plates were edge-supported in V-groove fixtures: the width/thickness ratios of the plate ranged from 15 to 60. Combinations of average stress, temperature and time producing given amounts of creep strain or failure are shown on a series of master curves, and the test results are compared with plate-strength and creep-failure stresses determined from semi - empirical approximations.

High-Temperature Embrittlement of Alloys and Steels

J. GLEN: 'A New Approach to the Problem of Creep.'

Jnl. Iron and Steel Inst., 1958, vol. 189, Aug., pp. 333-43.

Because the basic mechanism of creep is not fully understood, investigation of the creep properties of industrial alloys and steels has been carried out on an empirical basis. Extrapolation of data from short-time creep tests to times of, say, 100,000 hours has proved of doubtful reliability, and, without some theoretical justification, may lead to serious error. With these difficulties in mind, the author has sought a new approach to the subject, which has involved study of the high-temperature properties of a variety of steels (see, for example, abstract in *Nickel Bulletin*, 1957, vol. 30, No. 7-8, p. 122). A number of strain-age-hardening phenomena were observed, each associated with the presence of a particular alloying element. It has now been found that related effects can be detected in the creep curves of various steels and alloys. The present paper is limited to a simple statement of this new approach and to discussion of its application to the practical problem of extrapolation of creep data.

The strain-age-hardening phenomena are manifested by transitions in the creep rate, which are shown to the best advantage when the creep data are plotted in the form of log. strain/log. creep-rate curves. A series of such curves, obtained either at constant stress or at constant temperature, forms a regular pattern, so that similar curves can be estimated for lower stresses or temperatures. By integration, ordinary strain/time creep curves can then be obtained, and accurate extrapolation of creep or rupture tests can be made.

The results of tests on several manganese steels are used to illustrate the phenomena involved. In an appendix the author outlines the procedure employed to plot and integrate strain/creep-rate curves.

J. GLEN: 'Ductility in High-Temperature Rupture Tests.'

Ibid., vol. 190, Sept., pp. 30-9.

The problem of high-temperature embrittlement in alloys and steels (manifested during long-time rupture tests by failure associated with low elongation) has been the subject of much research. Although some light has been thrown on the mechanism of fracture of pure metals at high temperature, no theory has yet been advanced to explain the low ductility exhibited by more complex materials during failure.

The paper opens with a critical review of the present state of knowledge and, by way of illustrating the phenomena involved, presents and discusses the results of creep-rupture tests on the nickel-chromium-base alloy 'Nimonic 80A', a copper-beryllium alloy, the nickel-chromium-cobalt steel 'G 18B', and the following types of low-alloy steel: manganese, chromium-molybdenum, molybdenum-vanadium-titanium.

As a result of observation of the behaviour of these materials, the author outlines a tentative mechanism which seeks to account for (1) the phenomenon of intercrystalline failure during a transition in creep rate, and (2) the rôle of one alloying element in minimizing the embrittling effect of another.

J. GLEN: 'The Effect of Alloying Elements on Creep Behaviour.'

Ibid., Oct., pp. 114-35.

Development of new high-temperature alloys is contingent, *inter alia*, on a better understanding of the metallurgical changes which occur during creep

testing and, in particular, on determination of the influence of alloying elements in this connexion. The stress and temperature used in the test are important, since they govern the rate at which metallurgical changes take place; the initial condition of the specimen is also of significance. These factors are studied in the present paper.

The first part of the paper comprises a detailed exposition of the theory which the author advances in explanation of the effect of strain-age hardening or strain-induced precipitation on the creep behaviour of complex steels and alloys. This theory, which takes into account the influence of variation in composition, temperature, stress and heat-treatment, is illustrated by reference to log. strain/log. creep-rate curves.

In the second part of the paper creep data are presented in corroboration of the author's theoretical conclusions. The results reported (in the form of strain/creep-rate curves) indicate the effects of alloying elements and the relationship between stress, temperature and time of testing. The influence of nitrogen, carbon, manganese, chromium, molybdenum, vanadium, titanium and silicon contents, on the creep behaviour of ferritic steels is illustrated, and in other sections the author's theory is shown to be relevant also to materials with face-centred-cubic and close-packed-hexagonal lattice structures.

Austenitic steels of the 18–8 type, with or without titanium or niobium additions, other chromium-nickel austenitic steels ('G 18B', and A.I.S.I. types 326 and 327) containing molybdenum, cobalt or copper, an 80–20 nickel-chromium alloy and 'Nimonic 80A' are discussed as representative of materials of face-centred-cubic structure. Transitions in creep rate are shown by strain/creep-rate curves to occur in all these materials, though in most cases the particular constituent responsible for each transition is not known. (It is thought that tests on simple austenitic materials with single alloy additions would be of value in this connexion.) As with ordinary steels, it should be possible, from a series of strain/creep-rate curves, to predict the long-time creep properties of austenitic steels.

Oxidation-Resistance and Workability of Molybdenum-Nickel Alloys

E. S. BARTLETT, D. N. WILLIAMS and R. I. JAFFEE: 'Molybdenum-Nickel Dispersion Alloys.'

Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers, 1958, vol. 212, Aug., pp. 458–63.

Investigations carried out at Ohio State University and at Battelle Memorial Institute have indicated that the attainment of a truly oxidation-resistant molybdenum-base alloy for high-temperature service, with retention of high-temperature strength and toughness, is unlikely to be realized (see, for example, reports referred to in *Nickel Bulletin*, 1956, vol. 30, Nos. 3 and 5, pp. 45 and 93).

Among the more promising alloys which had been studied were certain compositions in the molybdenum-nickel system. These showed good oxidation-resistance at temperatures well above the normal melting point of MoO_3, because of the regenerative formation of protective nickel-molybdate ($NiMoO_4$) on the surface of the alloy during exposure at high temperatures. The usefulness of such materials is, however, limited by the fact that the alloy content required to obtain oxidation-resistance far exceeds the limits of the narrow terminal solid-solution boundary, and closely approaches the peritectic composition involving the brittle intermetallic compound MoNi. Because of the peritectic reaction, alloys made by melting and casting are brittle, since the MoNi phase, being the last to form, tends to occur as a continuous grain-boundary network round each molybdenum grain. An added problem is that the protective nickel-molybdate layer formed at high temperatures is unstable, undergoing one or more allotropic modifications during cooling. The volume changes associated with this modification are such as to cause violent splitting off of the coating from the surface of the metal, thus inhibiting protective effect during cyclic heating and cooling.

This later research was undertaken with a view to overcoming these problems, primarily by controlling the amount, size and location of the MoNi phase. By using powder-metallurgy techniques, and sintering the blended alloy-powder mixtures at a temperature below that of the peritectic reaction, it was found possible to produce molybdenum-nickel alloys with a randomly dispersed MoNi phase. Other materials used, in addition to binary molybdenum-nickel alloys, included high-molybdenum alloys containing (in addition to nickel) substantial amounts of chromium or small amounts of silicon or boron. (The latter elements had previously been shown to exercise a stabilizing influence on nickel molybdate.)

Details are given of methods used in preparation of the specimens, of the structural characteristics of the compacted and sintered alloys, and of oxidation tests at 1800°F. (982°C.) in dried air flowing past the specimens at 6·8 in./sec. Structural examination included, in addition to general metallographic inspection, a study (on the 10 per cent. nickel alloy) of the effect of dispersion distribution on oxidation behaviour. Fabricating properties were assessed by testing the hot rollability of bare specimens and of samples clad with nickel to secure improved distribution of the rolling stresses.

Results and Conclusions

Alloys prepared by addition of nickel, *viâ* MoNi or metallic nickel, showed oxidation-resistance superior to that of the ternary alloys. No tendency towards structural stabilization of the molybdate by the tertiary additions, in the amounts present, was noted. Marked lack of densification in the chromium-containing alloy during sintering, and the consequently porous nature of the specimen, rendered impossible any conclusion with reference to the

influences of chromium additions on oxidation behaviour.

Comparison of the oxidation-resistance and other characteristics of molybdenum-base alloys containing 5, 10, 15, 20, 25 and 30 wt. per cent. nickel (added as −325 mesh MoNi compound) showed that the 10 per cent. of nickel was the lowest alloy content conferring any improvement in oxidation-resistance over unalloyed molybdenum. The 20 per cent. nickel alloy was oxidation-resistant after formation of the molybdate coating, but this alloy did not possess the required dispersion qualities: even with 15 wt. per cent. nickel there was appreciable agglomeration of the dispersoid. For these reasons major interest was centred on the 10 per cent. nickel alloy: by comparison with unalloyed molybdenum, this material showed 100 per cent. improvement in oxidation-resistance.

The curves indicate that the characteristic oxidation of powder-metallurgy molybdenum-nickel alloys consists of two parts: (1) initial linear oxidation, at a rate about equal to that of unalloyed molybdenum, terminated by (2) change to a much slower oxidation rate. The transformation from the first portion to the second may be attributed to the formation of a layer of nickel molybdate of sufficient thickness and continuity to be semi-protective.

Although, by the rolling test, the alloys examined were rated as 'non-fabricable', the molybdenum-nickel specimens showed a considerable degree of hot toughness. It is believed that breakdown was the result of diffusion of nickel into the molybdenum-rich matrix material and was not attributable to the dispersion structure as such. Since the mechanism of failure was thus presumed to be by shear of the matrix material, it appears possible that by using fabrication methods which would minimize shear stress, dispersion alloys might fabricate to give good-quality finished material.

The overall conclusion drawn by the authors is that 'the observations made and the conclusions drawn do not preclude the further development of alloys which may have strength and toughness comparable to that of molybdenum at high temperatures, but whose oxidation-resistance may be significantly superior to that of molybdenum. The use of such alloys in place of molybdenum might have merit in applications in which the part is protected by cladding. Failure of the cladding would mean very rapid destruction of molybdenum, but by using a semi-oxidation-resistant molybdenum alloy, time of deterioration might be increased from a matter of minutes to several hours.'

Protection of Molybdenum by Coating

(I) Chromium-Nickel Coatings

D. E. COUCH, H. SHAPIRO, J. K. TAYLOR and A. BRENNER: 'Protection of Molybdenum from Oxidation at Elevated Temperatures.'

Jnl. Electrochemical Soc., 1958, vol. 105, Aug., pp. 450–6.

The authors demonstrate the possibility of protecting molybdenum by composite coatings of chromium+nickel. Details of experimental technique include the following points of particular interest:

Good adhesion of chromium to molybdenum is obtained by etching the basis metal with Murakami's solution and modifications of it, but such solutions were not suitable for molybdenum alloys containing titanium. Satisfactory results can be consistently obtained, however, for all types of molybdenum-base material tested, by anodic etching in a sulphuric-acid/phosphoric-acid solution (1 : 1 by volume) for 2–3 minutes at 2–10 amp./dm.^{2}.

Chromium was deposited from a conventional chromic-acid solution containing (g./L.) CrO_3 250 and H_2SO_4 2·5. The chromium was deposited under three conditions: (1) low-contraction chromium, deposited at 80–120 amp./dm.^{2}, at 85°–95°C., gave excellent adhesion; (2) satisfactory adhesion was obtained also by deposition, at 50°C., of bright chromium, at 20–30 amp./dm.^{2}; (3) chromium deposited at 25°C. gave poor coverage, and adhesion was unsatisfactory. The deposits made at these low temperatures tended to crack and to peel from the molybdenum base.

After chromium plating, the specimens were etched in 1 : 1 hydrochloric acid until the surface appeared grey. A nickel strike was then applied from an all-chloride bath (20–60 amp./dm.^{2}, for 1–3 min.), and finally plating was with nickel from an all-chloride or a Watts nickel solution.

Oxidation life was rated by the time for which the specimens remained intact, from the moment they were placed in the test furnace to the first moment at which molybdic-oxide smoke appeared or green nickel molybdate was observed.

Electrodeposited radioactive chromium was used as a means of detecting voids or inclusions in the nickel deposits, but this method of inspection was only partially successful. A satisfactory method of assessing the quality of the coatings was by heating the plated panels to 900°C. in an atmosphere of helium or hydrogen, and inspecting them for cracks or blisters in the coating.

Preliminary oxidation tests were made at three temperatures, 980°, 1100° and 1200°C.; the results indicated that the 1200°C. condition was too severe to be reasonable, and 1100°C. was therefore used in most of the tests. The oxidation-test results are evaluated in terms of effect of coating thickness and of temperature on life. The effect of mechanical working of the deposits before exposure was also studied.

It was found that a coating of 0·001 in. of chromium, followed by 0·007 in. of nickel, gave protection at 980°C. for 1200 hours; at 1100° for 500 hours, and at 1200°C. for 100 hours. Shot peening or ball milling of the coatings before test failed to give any increase in oxidation life. Study of the nature of the oxidation and diffusion phenomena occurring showed that both mechanisms contribute to failure.

The paper closes with a comparison of the actual life of the plated panels and the oxidation rates of

nickel reported in the literature, thus giving actual *vs.* theoretical oxidation life.

(*II*) *Nickel-Aluminium-Alloy Coatings*

D. E. COUCH, H. SHAPIRO and A. BRENNER: 'The Use of Nickel-Aluminium-Alloy Coatings for Protection of Molybdenum from Oxidation.'
Jnl. Electrochemical Soc., 1958, vol. 105, Aug., pp. 485–6.

In this paper the authors describe production of nickel-aluminium coatings by plating molybdenum with 25μ (1 mil) of chromium, followed by 175μ (7 mils) of nickel, and finally with 50–75μ (2–3 mils) of aluminium.

Several methods of forming nickel-aluminium-alloy coatings were studied, but some were found unsatisfactory. Plating from the hydride bath gave only poor adhesion of the aluminium to the nickel and the deposits tended to blister during heating at 600°–700°C. (the treatment used to alloy the aluminium with the nickel). Low-temperature fused-salt baths were also tried, but the limiting thickness of aluminium which could be obtained before treeing and rough deposits occurred was only about 12μ (0·5 mil). The most effective method of forming the nickel-aluminide layer was by electrodeposition from a fused-cryolite bath operated at 1000°C.: at current densities of 10–20 amp./dm.^{2} aluminium was deposited at sufficiently low rates to allow complete alloying with the nickel.

Tests on plated molybdenum panels showed that with coatings of 12μ (0·5 mil) or less of aluminium, oxidation took place at about the same rate as on electrodeposited nickel coatings: the oxidized coatings acquired the vitreous black nickel-oxide coating typical of nickel oxidized in air at 1100°C. With thicknesses of aluminium between 12μ (0·5 mil) and 20μ (0·8 mil) the rate of oxidation during the first 24–48 hours was markedly reduced: the panels assumed a bluish coating, which gradually turned black after about 100 hours of oxidation. Samples coated with a thickness of alloy equivalent to 50μ (2 mils) of aluminium, oxidized for 400 hours at 1100°C., developed white- or tan-oxide films, and showed weight gains of only 0·005 g./cm.^{2}. (Nickel-plated panels used as controls gave weight gains of 0·04 g./cm.^{2}.)

The experiments showed that the aluminide layer conferred significant protection on the molybdenum base, and it is probable that development of some method of reducing the rate of diffusion of aluminium into nickel would improve life, since it would maintain the aluminium concentration at a higher level in the surface for a longer period of time. It is possible that this might be accomplished by using a layer of nickel 75–125μ (3–5 mils) over some metal which would act as a diffusion barrier to the aluminium. Chromium might be suitable for this purpose. By this means the nickel-aluminide composition could be controlled at the optimum oxidation-resistant ratio.

Nickel-Chromium Electrical-Resistance Alloys

'High – Nickel – Chromium Alloys for Electrical Resistances.'
Aciers Fins et Spéciaux, 1958, No. 29, July, pp. 67–73.

The series of nickel-chromium and nickel-chromium-iron electrical-resistance alloys currently available for use as furnace elements are capable of satisfying a wide range of temperature requirements. Optimum use of these alloys is, however, contingent on observance of certain precautions. This review refers to alloys available, discusses service conditions which are known to be deleterious and, as a guide to the selection of the most suitable alloys for specific applications, outlines the general characteristics of the various grades.

Data are tabulated on the properties of the nickel-chromium and nickel-chromium-iron alloys discussed, and reference is made to accelerated testing and the relevance of the results obtained to actual service life. The effective life of a resistance alloy cannot be defined *a priori*, since it depends to a large extent on the service conditions. The nickel-chromium-base materials are, for example, susceptible to selective oxidation at the grain boundaries in a slightly oxidizing atmosphere: a strongly carburizing environment, or one containing even small amounts of sulphur, can also be extremely deleterious. The main causes of such contamination in furnace atmospheres are reviewed. Attention is drawn also to the importance of ensuring (for example, by proper ventilation) that the elements do not become overheated. In this connexion consideration is given to methods of determining relevant dimensions for wire and strip elements, in relation to the temperature required and the operational conditions.

A brief review is given of the advantages and limitations of the nickel-chromium and nickel-chromium-iron alloys: mention is made of the service temperatures for which they are suitable and conditions which might prove adverse.

Finally, the electrical characteristics of resistance alloys in the form of wire are correlated with variation in dimensions, and recommended sizes are given for strip.

Materials for Aerodynamic Applications

AMER. SOC. METALS: 'Metals for Supersonic Aircraft and Missiles.'
Proc. Conference on Heat-Tolerant Metals for Aerodynamic Applications, held Jan. 1957, under joint sponsorship of the American Society for Metals and the University of New Mexico.
Published by *Amer. Soc. Metals, Cleveland, Ohio*, 1958; 432 pp. Price $7.50.

The introductory note, by W. M. CANTERBURY, briefly outlines the problems arising from the most recent developments in supersonic aircraft and missiles, with particular reference to the need for development of improved materials which will withstand the combination of heat and mechanical stress involved in such service.

The papers contributed dealt with the aspects indicated below. All contained much detailed information, culled from published and unpublished sources, and correlated in such a way as to focus attention on the major problems involved and the methods of approach which may help towards solving them.

P. W. ROWE: 'Problems Relating to the Need for Heat-Tolerant Materials in Aerodynamic Applications', pp. 4–47.

Consideration of the thermal problems associated with high-speed flight, by means of examination of the thermal phenomenon, and a discussion of the manner in which high temperatures influence the design of aerodynamic structures. The reaction of materials to pressure, airflow, heat, and the composition of the service atmosphere is considered, as a basis for determining the requirements which should be specified for materials forming part of the integral structure of units operating under supersonic-flight conditions. The sphere of application of metallic materials is evaluated in the light of these requirements.

L. LUINI: 'Materials Property Requirements and Test Methods for Aerodynamic Applications', pp. 48–95.

Discussion is confined to a general review of the major properties required in materials which can be considered for service in this field, and of the test methods used for assessment of their suitability. The properties and methods reviewed are indicated below.

Characteristics	*Test Methods*
Room-temperature properties	Tensile Hardness Impact Metallographic Fatigue
Load-carrying ability	High-temperature tensile Creep Rupture Relaxation Fatigue Combined-stress
Surface stability	Oxidation Corrosion Abrasion and erosion
Structural stability	Room-temperature properties after exposure to high temperature Metallographic Long-time high-temperature
Special service conditions	Full-scale Component Rig test, simulating service conditions

The paper includes information on properties of typical high-temperature materials, as assessed by one or more of the methods referred to above. Data on nickel- and cobalt-base alloys form the major item in this section.

J. T. WABER: 'Kinetics of Oxidation in a Gas Stream', pp. 96–164, 166–9; disc. pp. 164–6.

It is pointed out that, in considering the effects of aerodynamic heating on structures, the oxidation behaviour of metals and alloys is of some significance. This factor, although at present relatively unimportant, will increase as velocities approach and exceed ten times the speed of sound, and skin temperatures become so high that evaporation of metallic materials and decomposition of refractories will occur unless strong cooling is applied internally. In the light of probable future conditions, the author discusses details of processes, such as ionic diffusion, which occur in oxide films during high-temperature oxidation, and briefly considers the magnitude of temperatures and heat fluxes which are likely to result from aerodynamic heating. The review covers oxidation behaviour and aerodynamic heating, considering the individual factors which are of importance in both respects.

C. L. FAUST: 'Protective Metallic Coatings', pp. 170–201.

Factors which determine the ability of coatings to provide the desired protection are considered, on the basis of laboratory and service tests. Methods which have been developed for application of oxidation-resistant coatings and the metals which have been deposited by the respective techniques, are tabulated, and information which has been gradually accumulated on the high-temperature oxidation-resistance and other properties of various types of coating is summarized, with references to original reports.

R. R. KENNEDY: 'The Light Metals Aluminium, Magnesium, Titanium', pp. 202–16.

Although for many applications in supersonic aircraft conditions would preclude the use of light metals and alloys, such materials will continue to be employed for some purposes, because certain types of unit will travel at supersonic speeds but at only low Mach numbers. In these conditions the service temperatures are well within the tolerance limits of light metals, thus making possible utilization of the advantage of low weight. The relative abundance of the light structural metals is a further advantage, from the economic standpoint. In this context, the properties of aluminium, magnesium and titanium are included in the subject matter of the Conference. Typical properties are reviewed in relation to the fields of service envisaged.

J. T. MILEK and B. L. MOLANDER: 'A.I.S.I. and Stainless Steels for Aerodynamic Applications', pp. 217–33.

Selection, by missile designers, of precipitation-hardenable austenitic steels of the '17-7 P.H.' type for skin and structural components has necessitated a comprehensive series of tests under conditions pertinent to such service. This paper reports results of investigations by North American Aviation,

Downey, California. Processes and thermal treatments used on such material and on '19-9 DX' (tungsten-containing chromium-nickel steel) are detailed, and mechanical and physical properties of the materials (at normal and at elevated temperatures) are recorded, illustrating the influence of heat-treatment and the characteristics required by the aircraft designer. Reference is made to tests now in progress to evaluate the qualities of other steels (among which are 'A.M.350', 'A.M. 355' and 'P.H. 15 - 7Mo') with respect to properties relevant to use of the materials in supersonic aircraft.

F. S. BADGER and G. A. FRITZLEN: 'Nickel- and Cobalt-base Alloys', pp. 234–57; disc., pp. 257–9.

See also Appendix I, pp. 363–4.

The important contribution which nickel- and cobalt-base alloys have made to recent progress in the aircraft field is briefly emphasized, as exemplified by typical materials such as the 'Nimonic' alloys, the 'Hastelloy' group, 'Multimet' alloy, 'Inconel' and 'Inconel X'. The major part of the paper is concerned with a review of data on nickel- and/or cobalt-containing wrought materials which, in sheet form, may contribute directly to the solution of some aerodynamic problems. Characteristics of representative alloys are discussed, with tables and graphs illustrating thermal expansion, thermal conductivity, resistance to thermal shock, oxidation-resistance, resistance to carburization, density, emissivity and other physical properties. The mechanical properties of the alloys, at normal and at elevated temperatures, are also summarized. An appendix gives data on effect of irradiation on hardness and on mechanical properties. In connexion with fabrication and thermal treatment, information is given on ageing characteristics and resistance to low-temperature embrittlement. Finally, some of the more interesting uses for sheet materials of the 'super-alloy' types are listed, with particular reference to the use of such materials in turbojet engines, ramjets, rocket-powered aeroplanes, missiles and nuclear power units. The information given in the paper is compiled from data supplied by a number of producers and users, to whom individual acknowledgement is made.

B. A. ROGERS: 'The Properties of Eleven Less Common Metals', pp. 261–314.

See also Appendices II and III, pp. 365–8.

Condensed, but detailed and well documented review of the properties of the following metals: chromium, niobium, hafnium, iridium, molybdenum, osmium, rhenium, tantalum, tungsten, vanadium and zirconium. Advantages and limitations, in relation to use in supersonic aircraft, are critically considered.

H. H. HAUSNER: 'Fundamentals of Metal-Ceramic Combinations (Cermets)', pp. 315–39.

The chapter deals with combinations of metals and metal compounds which cannot be fabricated other than by powder metallurgy, and with the application of that technique for the study of the solid-state phenomena which are responsible for the bond between the metallic and non-metallic components of cermets.

M. F. JUDKINS: 'Specific Ceramics and Cermets of Promise', pp. 340–52.

This paper collates recent work on some of the most promising types of cermet, summarizing properties in relation to intended applications. The tabular data include information on oxides, carbides, borides, nitrides, silicides, titanides, cermets and intermetallic compounds, and reference is made to the use of ceramic-metal coatings for protection against high-temperature deterioration.

Chapter 12 of the volume, compiled by J. R. TOWNSEND, comprises a review, summary and evaluation of the information contained in the foregoing papers.

A. V. LEVY: 'Performance of Materials at Elevated Temperatures for Aircraft and Missile Applications', pp. 369–99.

This chapter, forming Appendix IV, is concerned with temperature effects caused by aerodynamic heating of aircraft and missile structures. A set of curves shows the total temperatures due to aerodynamic heating at Mach numbers for altitudes from 10,000 to 60,000 ft., indicating the rapid increase of temperature associated with relatively small changes in Mach number and altitude. Major factors affecting the choice of materials working in extreme-temperature ranges are listed and data are tabulated on the elevated-temperature characteristics of aluminium- and magnesium-base alloys, steels (low- and medium-alloy types), normal and age-hardenable stainless steels, titanium alloys, high-temperature alloys of nickel- and cobalt-base types, molybdenum alloys.

Appendix V contains brief notes, by various authors, designed to give 'A General Review of the Current Position on Aircraft Alloys for Thermal Flight up to 1200°F.', pp. 400–23.

These notes were presented at a meeting sponsored by the American Society for Metals during the Western Metals Congress, March, 1957.

High-Strength Steels for Aeronautical Applications

See abstract on p. 326.

Lap Seam Welding of Precipitation-Hardening Nickel-Chromium Steel

E. J. FUNK and A. C. WILLIS: 'An Investigation of Lap Seam Welds in '17-7 P.H."

Welding Jnl., 1958, vol. 37, Sept., pp. 897–905.

The authors point out the potential usefulness of the precipitation-hardening stainless steels in the

aircraft industry, as exemplified particularly by '17-7 P.H.', but emphasize that full utilization of this potential is contingent on availability of a satisfactory welding technique.

The paper is concerned with two aspects of lap seam welding:

(1) Description of a method for measuring the secondary current in a seam welder with direct-energy three-phase primary, and for calibrating the measuring equipment;

(2) The seam welding of '17-7 P.H.' steel which is to be used in the TH-1050 condition. (As a basis to this section, some account is given of the heat-treatment cycles used for '17-7 P.H.' and of the properties and structure of the steel in various conditions.)

The descriptions of both the welding equipment and the properties and structure of welds made by it are well illustrated.

Complex Nickel-Molybdenum-base Alloys

O. PRESTON, C. F. FLOE and N. J. GRANT: 'Development and Testing of Air-Melted Nickel-Molybdenum Alloys with Minor Alloying Additions. Final Report.'
Oak Ridge Nat. Laboratory, Report 2520, June 16, 1958; 39 pp.

Severe embrittlement of vacuum-melted binary nickel-molybdenum alloys containing 17-36 per cent. molybdenum had been reported in *O.R.N.L. Report* 1990 (see abstract in *Nickel Bulletin*, 1957, vol. 30, No. 6, p. 99) and the influence of chromium, iron, vanadium and niobium in decreasing such embrittlement was dealt with in *O.R.N.L. Report* 2181 (see *Nickel Bulletin*, 1957, vol. 30, No. 7-8, p. 131).

The compositions of the alloys examined in the work now reported are given in table I below.

Bar stock from these experimental heats was received as ½-in. 12-mm. hot-rolled rounds. Solution treatment was for one hour at 2000°F. (1093°C.), followed by air cooling. Two specimens of each alloy were then aged for 100 hours at 1300°F. (705°C.), together with specimens of each of the five alloys used in the previous investigations. Test bars from these specimens (together with specimens of commercial 'Hastelloy B') were tested in stress-rupture at 1300° and 1500°F. (705° and 815°C.) at the stress for 100-hour rupture life which had been determined for 'Hastelloy B' from published data.

Stress-rupture tests were made at 1350°, 1500° and 1650°F. (732°, 815° and 898°C.) for each of the six alloys, in two conditions of heat-treatment, under stresses chosen to give rupture lives in the range 1-500 hours. Alloys 'INOR-1' and 'INOR-2' were tested in the solution-treated condition and after ageing for 16 hours at 1500°F. (815°C.). Alloys 'INOR-3, -4, -5 and -6' were tested after solution treatment at 2000°F. (1093°C.) followed by ageing for 16 hours either at 1300°F. (705°C.) or at 1500°F. (815°C.).

A detailed report is made on the stress-rupture tests, and these data are correlated with photomicrographs showing the structure of the alloys in various conditions.

The results show that the addition of minor amounts of other alloy elements is effective in eliminating the embrittlement observed in vacuum-melted nickel-molybdenum alloys. In the absence of ageing additions, the presence of 5 per cent. of chromium brings the room-temperature strength of the 16 per cent. molybdenum alloy up to that of the 20 per cent. molybdenum binary alloy, but chromium does not effectively replace molybdenum in improving rupture strength.

Addition of 1 per cent. of aluminium and 1·6 per cent. of titanium results in effective ageing at 1300°F. (705°C.), but at 1500°F. (815°C.) the alloy overages. The ageing has only a slight effect on stress-rupture properties in the range 1350°-1650°F. (732°-898°C.). Increasing the aluminium content to 2 per cent., with a combination of 1·6 per cent. of titanium, significantly improves the stability of the ageing reaction with respect to both room-temperature strength and elevated-temperature stress-rupture

Table I

Composition of Nickel-Molybdenum-base Alloys

Alloy	C %	Mn %	Fe %	S %	Si %	Cu %	Ni %	Cr %	Al %	Ti %	W %	Mo %	Nb %
'INOR-1'	0·01	0·47	0·28	0·002	0·47	0·02	78·44	—	—	—	—	20·29	—
'INOR-2'	0·04	0·54	0·69	0·002	0·52	0·02	76·72	5·28	—	—	—	16·17	—
'INOR-3'	0·01	0·53	0·18	0·001	0·57	0·02	79·86	—	1·08	1·62	—	16·11	—
'INOR-4'	0·01	0·49	0·49	0·001	0·53	0·02	77·83	—	1·96	1·68	—	16·97	—
'INOR-5'	0·10	0·94	1·66	0·002	0·68	0·02	78·59	—	—	—	2·69	13·15	2·15
'INOR-6'	0·02	0·57	0·59	0·003	0·62	0·02	74·01	5·30	1·15	1·69	—	16·01	—

properties: this is particularly noticeable at 1650°F. (898°C.). At the lower aluminium content (1 per cent.) the addition of 5 per cent. of chromium results in a marked increase in response to, and stability of, the ageing reaction, with respect to both room-temperature properties and stress-rupture properties in the range 1350°–1650°F. (732°–898°C.). This modification is not, however, so effective at 1650°F. (898°C.) as that achieved by increasing the aluminium content.

Addition of carbon 0·1, tungsten 2·7, niobium 2·2, per cent. effected no ageing response.

Consideration of the mechanical properties at high temperatures, and of the microstructures of the alloys, indicates clearly that the benefits of ageing due to titanium+aluminium are not so great in nickel - molybdenum - base alloys as in nickel - chromium-base types such as the 'Nimonic' series and 'Inconel X-550'.

High-Temperature Properties of Wrought Titanium-Stabilized Austenitic Steel containing Molybdenum

B. J. CONNOLLY and G. BOYD: 'The High-Temperature Properties of Four Wrought Steels used in the Chemical Industry.'

Instn. Mech. Engineers, Preprint, 1958; 15 pp.

Although austenitic chromium-nickel and low-alloy chromium-molybdenum steels find applications in the chemical industry, few data are available on two steels of particular interest in this respect: low-carbon 3 per cent. chromium-molybdenum steel and titanium-stabilized 18-10 chromium-nickel steel containing molybdenum. As a contribution towards filling the gap, creep and short-time high-temperature tensile tests were carried out at the I.C.I. Creep Test Station. The low- and medium-carbon grades of 1 per cent. chromium-molybdenum steel were also included in the test programme, to provide confirmatory data supplementing the extensive information already available on these types. The results of the authors' investigations are recorded in this paper.

Details of composition, heat-treatment and room-temperature mechanical properties are presented: data derived from the tests on each steel are given in tabular and graphical form. Photomicrographs of the steels, before and after testing, supplement discussion of the effects, on microstructure, of long-time heating under stress.

Short-time high-temperature tensile tests on the chromium-nickel stainless steel showed that strength and proof stresses fell steadily with rise in temperature up to 200°C., but then remained practically constant in the range 200°–500°C. Above 500°C. ultimate strength decreased sharply; proof stresses fell to a lesser degree. Proportional limit was relatively low at all test temperatures, and there was no pronounced decrease in the values obtained over the range room temperature to 800°C. The ductility of the steel deteriorated significantly with rise in temperature to 500°C., but then improved, to attain a very high value at 800°C.

Creep and stress-rupture tests were made at 500°, 600°, 700° and 800°C. The effects of creep became important in the region of 600°C., and at 800°C. the steel exhibited appreciable creep at stresses greater than 1 t.s.i. (1·5 kg./mm.^{2}). Ductility decreased as the time of exposure was lengthened, but at lower test temperatures it remained constant for longer periods.

The high-temperature tests were utilized also as a means of determining the effects, on the constitution and properties of the steel, of long-time exposure at the test temperature. The results of room-temperature mechanical tests on broken creep-test specimens of the chromium-nickel steel, and of X-ray-diffraction examination, are presented in the form of an appendix to the paper. Exposure at elevated temperature was found, in nearly all cases, to result in a slight increase in tensile strength, a finding which coincides with the increase in hardness caused by formation of sigma phase. Ductility decreased as the quantity of sigma phase increased. A notable feature of the tensile results was the very high yield points obtained, particularly with specimens broken at 500° and 600°C.

The amount of sigma phase present was governed by temperature of exposure and time at temperature. At 800°C. transformation of all the ferrite to sigma and austenite was completed in less than 245 hours; at 700°C. the time required was about 2,000 hours, and at 600°C. about 6,000 hours. Creep cracking was often associated with the presence of sigma phase, although in the stress-rupture specimens such cracking occurred only when the total amount of deformation involved was considerable. After testing at 500°C. all the specimens contained a very finely divided black precipitate in the ferrite phase: the precipitate (designated α') was found to correspond to α iron, but it was non-magnetic. It is considered that this phase is an intermediate transformation product between the ferrite and sigma: transformation to sigma at 500°C. would probably take many years and no sign of sigma was found even after 7,771 hours at that temperature.

Design stresses, based on the data obtained, are suggested for each of the four steels.

Effect of Cold Work on the Fatigue Limit of a Nickel-Manganese-Chromium Austenitic Steel

B. CINA: 'The Effect of Cold Work on the Fatigue Characteristics of an Austenitic Alloy Steel.'

Jnl. Iron and Steel Inst., 1958, vol. 190, Oct., pp. 144–57.

Increases in fatigue limit resulting from cold work have been reported for plain-carbon steels, non-ferrous metals, titanium alloy and stable and unstable austenitic steels, though, in the case of the stable austenitic steel (15–28 chromium-nickel material), a definite increase was determined only after 27 per cent. elongation. In none of the investigations was any attempt made to explain the magnitude of the

increase in fatigue limit in relation to the increase in static tensile properties produced by cold work. In this paper the author records the results of reverse-stress tests on a steel of which the following composition is typical: carbon 0·63, silicon 0·41, sulphur 0·008, phosphorus 0·034, nickel 8·35, manganese 7·45, chromium 4·00, nitrogen 0·015. The M_s temperature was below −196°C., and no $\gamma \rightarrow \alpha$ transformation could be induced by cold working at room temperature, i.e., the steel could be considered stable at room temperature.

Specimens of the steel were recrystallized and softened by heat-treatment for half an hour at 1050°C., and then cold worked at room temperature, to achieve 0, 5, 10, 20 or 30 per cent. elongation. The fatigue specimens machined from the cold-worked tensile pieces were subjected to reversed-direct-stress and pulsating-tension fatigue tests. Attention was paid more to the actual fatigue limit than to the precise slope of the finite-life portion of the S/N curves obtained.

The fatigue experiments were so designed as to throw light on the following factors:

(1) The effect of cold work.

(2) The effect of mode of fatigue stressing.

(3) The effect of cold work followed by light tempering.

(4) Hardness changes during fatigue testing.

(5) and (6) Structural changes during fatigue testing, as detected by X-ray diffraction and optical microscopy.

(7) The possible rôle of creep in the pulsating-tension fatigue tests.

The results, which are reported at length, led to the following main conclusions:

'(1) It is suggested that, from the theoretical viewpoint, unfair criteria have hitherto been used for assessing the effect of cold work on fatigue strength. A clearer understanding is obtained (*a*) if the effect of cold work on static tensile properties (especially proof stresses) is compared initially with its effect on the fatigue limit determined dynamically in tension, and only thereafter with that on the fatigue limit determined dynamically in alternating stress; and (*b*) if the fatigue ratio is considered as the quotient of the fatigue limit and the yield point or a small proof-stress value.

'(2) Cold work has been found to be simultaneously beneficial and deleterious, the balance depending on the extent of cold work and the mode of stressing in fatigue. It is beneficial in that it restricts plastic deformation and so hinders fatigue damage. It is deleterious in that it introduces dislocations whose movement can lead to fatigue-damaged zones.

'(3) For an austenitic alloy steel, the effect of static cold work in tension was, for fatigue in pulsating tension, to give a fatigue ratio based on 0·2 per cent. proof stress of 1·0 for up to 10 per cent. cold work. Thereafter the fatigue ratio decreased, although it was still 0·74 after 30 per cent. cold work. For fatigue in alternating tension and compression a limited beneficial effect of cold work was found, the fatigue ratio dropping from 0·94 in the softened condition to 0·42 after 30 per cent. cold work.

'(4) The markedly inferior fatigue limits and ratios for alternating stress, as compared with pulsating tensile stress, were attributed to the operation of a Bauschinger effect in the former and not in the latter. This effect allows the dislocations introduced by the cold work to be utilized more readily to form a fatigue-damaged zone. The absence of a Bauschinger effect in pulsating tension allows the material to withstand higher stresses before sufficient plastic deformation can occur to form a fatigue-damaged zone. Supporting evidence for this is provided from hardness measurements and from metallographic and X-ray-diffraction examination.

'(5) Light tempering treatments after cold work, which removed the peak residual microstresses, failed to raise the fatigue limit in alternating stress.

'(6) Fatigue cracks were detected within 10 per cent., and only after 75 per cent., of the average lives in alternating stress and pulsating tension, respectively.

'(7) Fatigue cracks formed in alternating stress were found to propagate 100 to 200 times more readily by further cycles of alternating stress than by cycles of pulsating tensile stress of the same magnitude, i.e., crack propagation in alternating stress occurred by a mechanism probably akin to that of its formation, and was not solely dependent on the joint effects of stress concentration at the tip of the crack and the applied alternate tensile cycle.

'(8) A critical amount of static cold work was required before fatigue failure could be obtained in pulsating tension, and likewise of dynamic cold work for failure in alternating stress of initially softened material.

'(9) Creep may have been a factor in the pulsating tensile fatigue tests.'

Stress-Corrosion Cracking of Chromium-Nickel Austenitic Steels

H. L. LOGAN: 'Studies of Stress-Corrosion Cracking of Austenitic Stainless Steel.'

Welding Jnl., 1958, vol. 37, Oct., pp. 463s-7s.

Although operational experience has shown that corrosion cracking of austenitic stainless steels has occurred mainly as a result of exposure to hot dilute chloride solutions, in most investigations of the phenomenon use has been made of concentrated chloride test solutions. The initial phases of the author's study of the mechanism of stress-corrosion were therefore concerned with the development of a corroding medium which would be more representative of service conditions. The results showed that stress corrosion could be induced by exposure to a boiling solution containing 3½ per cent. of sodium chloride and 1 per cent. of ammonium

nitrite (see LOGAN and SHERMAN, *ibid.*, 1956, vol. 35, Aug., pp. 389s-95s; *Nickel Bulletin*, 1956, vol. 29, No. 10-11, p. 196).

The steels tested in the phase of the investigation reported in this paper were normal and low-carbon grades of chromium-nickel 18-8 stainless steel. The scope of the test programme is indicated below.

(1) For commercial reasons the sodium-chloride/ammonium-nitrite solution was subsequently modified, and investigation was made of the suitability, as a corrosive medium, of a boiling solution containing $0{\cdot}1N$ NH_4Cl, $0{\cdot}1N$ $NaNO_2$ and $0{\cdot}5N$ NaCl (which would be expected, after reaction, to give an effective solution of $0{\cdot}1N$ $NH_4NO_2 + 0{\cdot}6N$ NaCl). Later investigations were centred on the use, as a test corrosive, of a solution containing $0{\cdot}5N$ NaCl and $0{\cdot}1N$ $NaNO_2$.

Tensile tests were carried out on specimens which had been subjected, in the boiling sodium-chloride/ammonium-nitrite solution and in the boiling $NaCl/NaNo_2$ solution, for times up to 48 hours, to stresses equivalent to their room-temperature yield strength.

(2) HOAR and HINES (see abstract in *Nickel Bulletin*, 1956, vol. 29, No. 5, pp. 96-7) had reported that wire specimens exposed in boiling 42 per cent. $MgCl_2$ solution, but stressed only during the latter part of the exposure period, failed in approximately the same time as those that had been stressed throughout exposure. Since these data were of significance for the development of current theories of stress corrosion, their validity was checked in the present investigation by exposing specimens in boiling $NaCl/NaNO_2$ solution for 48 hours, and stressing them either for the first or the last two hours of exposure. The specimens were subsequently broken in tensile tests.

(3) To investigate the effect of corrosion products on crack propagation, corrosion tests were carried out, on unstressed specimens, in solutions of compounds which could be formed during chloride corrosion of steel ($FeCl_3$, $FeCl_2$, $FeCl_2 + HCl$, $NiCl_2$, $CrCl_3$ and $MnCl_2$).

(4) Determination of anodic and cathodic polarization curves for unstressed specimens in the boiling $NaCl/NaNO_2$ solution.

(5) The effects of strain were studied by determination of stress/strain curves for specimens immersed in boiling distilled water.

The results, correlated with the stages of the test programme numbered above, may be summarized as follows:

(1) Both of the solutions used in the investigation induced stress-corrosion cracking in the stainless steels investigated. The tensile strength and elongation of the specimens under load in the boiling $NaCl/NH_4Cl/NaNO_2$ solution decreased with increase in the exposure period. Nitrite-ion concentration of the solution also fell with time of exposure. The number of cracks per specimen increased rapidly for the first 4 hours of exposure and thereafter did not change appreciably with time.

Similar deterioration was observed in the tensile properties of steels exposed in the boiling $NaCl/NaNO_2$ solution. No decrease in nitrite-ion concentration was observed during exposure periods of 48 hours.

(2) Values for tensile strength and elongation of specimens loaded for the first two hours of exposure were: 71,500 p.s.i. (32 t.s.i.; 50 kg./mm.^{2}) and 16½ per cent. Specimens exposed only for the last 2 hours gave values of 85,700 p.s.i. (39 t.s.i.; 61·5 kg./mm.^{2}) and 52 per cent.

(3) Macroscopic evidence of corrosion was found only in those specimens exposed in $FeCl_3$ solutions having a pH of less than 5, and in the $FeCl_2 + HCl$ solution at a pH of 1·3.

(4) Accelerated stress-corrosion cracking was induced in the $NaCl/NaNO_2$ solution when the stressed steel was made the anode in an external circuit with a current density of 0·03-1 ma./cm.^{2}. From cathodic-polarization curves it was found that a current density of approximately 0·04 ma./cm.^{2} or more would cathodically protect the material against stress-corrosion cracking.

(5) The yield strength of the material was 5-15 per cent. lower at 100°C. than at room temperature. Elongation of specimens stressed to their room-temperature yield strengths continued for at least one hour after the stress was applied. The strain rate was non-uniform with time.

It is postulated that stress-corrosion cracking is electrochemical in inception and, in part at least, in propagation. Sporadic but repeated creep in certain favourably oriented regions ruptures the protective film on the metal in narrow areas normal to the applied stress. The film-free area constitutes a small anode; the cathode area is very large, and corrosion is rapid until the film re-forms. This eventually produces, in a direction normal to the applied stress, a condition of stress concentration which plays an important part in the propagation of stress-corrosion cracking.

Resistance of Unstabilized Austenitic Steels to Corrosion by Chloride-containing Steam Condensates

F. B. SNYDER, T. A. MCNARY and F. EBERLE: 'Investigation of Suitability of 18-8 (Type 304) Alloy for Superheater Service with Respect to Corrosion and Stress-Corrosion Behaviour in Chloride-bearing Steam Condensate.'

Amer. Soc. Mechanical Engineers, Paper 57-*A*-174, 1957; 17 pp.

Austenitic nickel-chromium steels of the niobium- and titanium-stabilized types have for many years been successfully used as superheater materials, but doubts have been expressed as to the advisability

of using the unstabilized grades, although from the economic point of view, and with regard to structure, they offer certain advantages.

The main objection raised has been that, after prolonged heating in the sensitizing range of temperature the straight nickel-chromium steels might prove more susceptible than the stabilized type to intercrystalline corrosion or stress-corrosion cracking when in contact with chloride-containing condensate.

This paper reports investigation of the comparative resistance of stabilized and unstabilized '18-8' chromium-nickel steels to synthetic solutions of chlorides of two different concentrations, and of the behaviour of unstabilized '18-8' steel in field-service conditions in a boiler.

The laboratory tests showed that in synthetic condensates containing 38·4 p.p.m. and 2,000 p.p.m. the unstabilized steels, like the stabilized types, do not suffer attack. Stress-corrosion tests in the same media confirmed that *all* the steels were susceptible to cracking: no significant difference was observed in this respect between stabilized and unstabilized types.

Unstabilized '18-8' steel installed in the pendant superheater of an operating stationary boiler showed no intercrystalline attack after 54 months of service.

Corrosion of Stainless Steel by Fused Sodium Hydroxide

G. P. SMITH and E. E. HOFFMANN: 'Corrosion Products formed in the Reaction between Fused Sodium Hydroxide and Iron-rich Alloys of Iron, Chromium and Nickel.'

U.S. Atomic Energy Commission, Oak Ridge Nat. Laboratory Report 2156, May 14, 1957; 8 pp.

This report is one of a series describing reactions between fused sodium hydroxide and alloys composed of elements which have widely differing reactivities in a hydroxide melt. Such reactions have been found to involve complex solid-state processes which give rise to selective leaching of the more reactive alloy components. The decrease in volume of the alloy associated with this leaching effect takes place by retreat of the alloy surface along preferred paths, to form a network of deep pits or channels extending inward from the alloy/hydroxide interface. For some of the materials studied it was found that these channels were filled with corroding medium, but for 'Inconel' they contained reaction products.

This third report relates to the microstructures of corrosion-product layers formed by the action of fused sodium hydroxide, at 815°C., on straight '18-8' chromium-nickel steel and titanium-stabilized '18-8' and also with tests of four high-purity alloys having the following nominal compositions: (*a*) iron 80, chromium 20, per cent.; (*b*) iron 80, chromium 10, nickel 10, per cent.; (*c*) iron 74, chromium 18, nickel 8, per cent.; (*d*) iron 60, chromium 20, nickel 20, per cent. The 'capsule-test' technique, described in earlier reports, was again used.

The metallographic evidence presented indicates that the reaction of these materials with fused sodium hydroxide is similar to that observed with 'Inconel'. For most of the materials, corrosive penetration proceeded more rapidly along grain boundaries than through grains: similar sensitivity of grain boundaries to selective attack had been observed in 'Inconel' exposed to sodium hydroxide under certain conditions, and in nickel-iron alloys in contact with gaseous oxygen. A noticeable microstructural feature of the corrosion-product layers formed on almost all the alloys was that a substantial portion of the inorganic reaction product was in the form of very slender particles. These results indicate that such shapes are not peculiar to an alloy of the 'Inconel' composition, but are of more general occurrence.

Behaviour of Metals in Contact with Fluorine

H. W. SCHMIDT: 'Compatibility of Metals with Liquid Fluorine at High Pressures and Low Velocities.'

Nat. Advisory Committee for Aeronautics, Research Memorandum E58D11, July 15, 1958; 14 pp.

Due to the high reactivity of fluorine, some difficulty has been experienced in systems handling this material. It is believed that the majority of failures occurring in such plant are due to one or more of the following causes: (1) choice of unsuitable materials of construction, (2) high flow velocity, with resulting turbulence and impact effects, (3) high pressure, (4) exposure of sharp edges or corners to the flow of fluorine, (5) conditions arising from high-velocity flow through pinhole leaks, (6) contamination, causing localized reactions.

The investigation reported was designed to establish the extent to which any of these conditions contributes to failure in fluorine systems.

Metals usually considered to be suitable for such applications were tested for compatibility under exposure to controlled fluorine conditions. Specimens of nickel, nickel-chromium stainless steel, aluminium and brass were exposed in forms representative of components commonly used in flow systems, and providing for simulation of potentially dangerous conditions which were to be studied.

The specimens were exposed to flow velocities up to 400 ft./sec. (120 m./sec.), at pressures up to 1500 p.s.i. (1 kg./mm.^{2}). Most of the tests were run with the apparatus submerged in liquid nitrogen at −320°F. (−196°C.), and some runs were made with samples of metal tube initially at ambient temperature. Apparatus, test conditions, and results are fully described.

None of the metal specimens eroded, decomposed or showed any measurable physical or chemical changes. In a run made with a 'Teflon' specimen, instantaneous chemical reaction and decomposition occurred.

See also following abstracts.

Nickel Alloys for Handling Fluorine and its Compounds

HENRY WIGGIN AND CO., LTD.: 'Wiggin Nickel Alloys *v.* Fluorine and Fluorine Compounds.'
Publn. 1554*, 1958; 40 pp.

The publication presents data on the corrosion-resistance, with respect to fluorine and its compounds, of the following materials produced by Henry Wiggin and Company, Ltd.: nickel, 'Monel', 'Inconel', 'Corronel B' and 'Ni-O-Nel'. A summary of industrial experience, collated from a variety of sources, indicates the usefulness of nickel-base alloys in this field.

The information given is similar in many respects to that contained in the brochure published by THE INTERNATIONAL NICKEL CO., INC., an abstract of which appeared in *Nickel Bulletin*, 1958, vol. 31, No. 7, p. 198.

Determination of the Die-Filling Characteristics of Stainless-Steel Powders

See abstract on p. 313.

Electrochemical Etching of Stainless Steels

Y. B. MALEVSKII: 'Reagent for the Electrolytic Etching of Chromium-Nickel Austenitic Alloys.'
Zavodskaya Lab., 1958, vol. 24, pp. 111–12.

The author describes a method of electrolytic etching of 18-8 and 25-20 chromium-nickel steels, using an electrolyte comprising a 30 per cent. aqueous solution of KCl, with a current density of 0·013–0·20 amp./cm.^{2}. The duration of treatment is 20–50 seconds. The effect is that austenite is dissolved, leaving carbides and sigma phase in relief. Typical results are illustrated by photomicrographs.

Nickel-Alloy Steels for Use in the Chemical and Petroleum Industries: B.S. Specifications

BRIT. STANDARDS INSTN.: 'Steels for Use in the Chemical, Petroleum and Allied Industries.'
B.S. 1501–1506: 1958; 84 pp. Price 25s.

This series of standards, which was prepared as part of a comprehensive scheme of codes for pressure vessels used in the chemical and allied industries, covers plates, bars, and sections; forgings; castings; and bars for bolting material. It supersedes the edition published in 1950. Each of the standards is in two sections: the first lays down general requirements in connexion with manufacture, analysis, testing, etc.; the second stipulates the composition, heat-treatment and mechanical properties required in the individual steels covered.

The standards and the types of steel to which they relate are indicated below:

B.S. 1501 'Carbon and Alloy Steel Plates, Sections and Bars.'

B.S. 1503 'Carbon and Alloy Steel Forgings.'

B.S. 1504 'Carbon and Alloy Steel Castings.'

B.S. 1506 'Carbon and Alloy Steel Bars for Bolting Material.'

Steels covered by the specifications:

Carbon Steels (various grades).

Manganese Steel (forgings, plates, sections and bars only).

Molybdenum Steel.

3½ per cent. Nickel Steel (not included in *B.S.*1506).

Chromium-Molybdenum Steel (various grades).

Chromium-Nickel Austenitic Steels.

(*B.S.* 1501, 1503 and 1504 refer to 18-8 type unstabilized grades; grades stabilized with niobium or titanium; grades containing 2½ per cent. molybdenum, with or without niobium or titanium additions; unstabilized grades containing 3½ per cent. molybdenum.

*B.S.*1506 covers four unstabilized grades of 18-8 type; two grades stabilized with titanium or niobium; a titanium-stabilized free-machining grade; a grade containing 2½ per cent. molybdenum.)

Three appendices to the publication give, respectively, details of the numbering system which has been adopted to indicate types of steel; existing British Standards which have been separately published for materials now included in the composite schedule, and the nearest equivalent American steel in either the A.S.T.M. or A.I.S.I. series; the procedure specified for welding repairs on castings covered by *B.S.* 1504.

It will be noted that no specifications relevant to the designations *B.S.* 1502 and 1505 have been included: these numbers are reserved for other forms of material which may be required as a result of future developments.

BRIT. STANDARDS INSTN.: 'Steels for Use in the Chemical, Petroleum and Allied Industries: Low-Temperature Supplementary Requirements to *B.S.* 1501–1506.'
B.S. 1510: 1958; 6 pp. Price 4s. 6d.

The standard, which is supplementary to those referred to in the preceding abstract, covers additional requirements for steels intended for operation under severe conditions at sub-zero temperatures (down to −190°C.) or for applications which necessitate a high degree of notch ductility. It is applicable to carbon- and alloy-steel plates up to 2-in. thick, and to forgings, sections and bars having a ruling section not exceeding 4 in. Requirements are laid down with respect to composition, heat-treatment, impact testing and impact properties.

Stainless-Steel Hypodermic Dental Needles

BRIT. STANDARDS INSTN.: 'Hypodermic Dental Needles.'
B.S. 2983: 1958; 11 pp. Price 4/-.

This specification is one of a series relating to hypodermic equipment, which has been prepared under the aegis of the Surgical Instruments and

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* *We shall be pleased to supply a free copy of this publication.*

Medical Appliances Industry Standards Committee.

Requirements are specified for needles of two types: interchangeable-head and cartridge. Dimensional and test requirements are laid down and two alternative angles of point are specified for each type and size of needle.

The blades of the needles are to be made either of martensitic stainless steel of one of the En 56 series, or of austenitic stainless steel of one of the En 58 series. Both groups are covered by *B.S.* 970. The chromium and nickel contents of the austenitic steels referred to fall within the ranges 17–20 and 7–12 per cent., respectively; molybdenum additions are specified for certain of the steels.

Welded Stainless-Steel Tubes: Specification

BRIT. STANDARDS INSTN.: 'Welded Austenitic Stainless-Steel Tubes for Mechanical, Structural and General Engineering Purposes.'

B.S. 3014:1958. Price 4s. 6d.

The Standard covers tubes manufactured from austenitic stainless-steel strip welded across the abutting edges throughout their length. Part I details general requirements (including the materials from which the tubes are to be made); Part II covers as-welded tubes, Part III cold-drawn welded tubes. Dimensional tolerances are laid down for both classes.

Six grades of steel are listed:

Grades 1 and 2 are unstabilized chromium-nickel steels, containing, respectively, carbon 0·08 max., with chromium 17·5–19·5 and nickel 8–10 per cent., and carbon 0·15 max., with chromium 17·5–19·5 and nickel 7·5–9·5, per cent.;

Grades 3 and 4 are titanium-stabilized '18–8' types;

Grade 5 is a niobium-stabilized type containing carbon 0·08 max., chromium 17–20, nickel 9–13, per cent.;

Grade 6 is a molybdenum-containing type: carbon 0·08 max., chromium 16·5–18·5, nickel 10·0–12·0, molybdenum 2·5–3·0, per cent.

Tests provided for in the specification include tensile, flattening and expanding tests, and determination of susceptibility to intercrystalline corrosion, by means of the acidified copper-sulphate test, on specimens which have been subjected to heating at specified temperatures. To test the quality of the welds, a hydraulic test of the tubes is also laid down.

Nickel-Aluminium-Bronze Marine Propellers

See abstract on p. 322.

Stress-Relief Treatment of Stainless Steel

R. A. HUSEBY: 'Stress-Relieving of Stainless Steels and the Associated Metallurgy.'

Welding Jnl., 1958, vol. 37, July, pp. 304s–15s.

This is one of the 'interpretive reports' issued under the aegis of The Welding Research Council. It contains a systematic analysis of the nature and origin of stresses which may be introduced into steel, and of the effect of stress-relieving treatments, as influenced by the composition of the steels and their inherent constitution and structure.

It is pointed out that in the austenitic stainless steels residual stresses introduced by fabricating operations, including welding, may adversely affect the serviceability of the materials in certain corrosive environments, and may also influence dimensional stability. The conventional stress-relief temperature is within the range which affects the microstructure of such steels, and may result in a lowering of ductility, impact properties and general corrosion-resistance. Stress-relief treatment of austenitic nickel-chromium steels is therefore usually applied only when the steel is to be used in service conditions involving severe corrosive attack or calling for a specially high degree of dimensional stability. Where it is necessary to apply such treatment, preference should be given to compositions which are relatively insensible to deterioration on heating in the stress-relief range.

Thermal stress relieving of the ferritic chromium stainless steels after fabrication is necessary to obtain the desired mechanical properties and corrosion-resistance. The microstructure resulting from welding or heating to intermediate temperatures is not conducive to good mechanical properties or corrosion-resistance; hence the necessity for stress-relieving or annealing after fabrication.

The martensitic stainless steels are highly hardenable. They require pre-heating and high interpass temperatures during welding, and immediate post-heating after welding to avoid cracking. The post-heating temperature is therefore usually that required for stress relief.

In welding the straight-chromium stainless steels thermal stress-relief treatment is mandatory under most welding codes, as necessary in order to obtain the required level of mechanical properties and corrosion-resistance.

The original report contains much supporting discussion, on which the above general conclusions and recommendations are based: it is supported by a selected *bibliography* referring to major literature.

High-Temperature Brazing Alloys for Joining 'Inconel' to Stainless Steel

D. A. CANONICO and H. SCHWARTZBART: 'Development of Oxidation- and Liquid-Sodium-Resistant Brazing Alloys.'

Wright Air Development Center, Tech. Report 57-648, Mar. 1958; 40 pp. *P.B.*131745.

The report, which is in two sections, presents the results of preliminary investigations carried out as part of a programme to develop alloys suitable for brazing 'Inconel' to 18–8 (Type 301) chromium-nickel stainless steel. Both materials were to be brazed in sheet form. The main requirements were: (1) high resistance to oxidation in air and to corrosion by liquid sodium at 1650°F. (900°C.); (2) a flow temperature in the range 1750–1900°F. (955–1035°C.);

(3) satisfactory flow characteristics; (4) sufficient ductility to permit forming and pre-placing in the joint. Details of the experiments made to satisfy these requirements are given in the first and main part of the report. The other phase of the investigation, the development of a simple method of determining the ductility of a brazed joint at the service temperature, is described in the second part.

In the light of the requirements of the investigation, three metals were selected as offering promise as bases for development of suitable alloys: iron, nickel and chromium. A study was then made of relevant binary phase diagrams, to determine the systems exhibiting eutectic reactions in the temperature range required. To those alloys with eutectics above the upper limit of 1900°F. (1035°C.) a third and, in some cases, a fourth and fifth element were added, as a means of lowering the temperature to the range required. The effect of alloying additions of germanium, silicon, indium, beryllium, antimony, titanium and lithium was studied. A total of 91 alloys (the compositions of which are given) were prepared for screening by means of melting and flow tests. Of these alloys, 16 satisfied the requirements and were deemed suitable for further evaluation. To determine oxidation-resistance T specimens brazed with each alloy were exposed in air for 500 hours at 1650°F. (900°C.): the same alloys were subjected to erosion tests, and tension shear properties were also determined. Under these conditions four compositions were found to exhibit the desired oxidation-resistance, i.e., they were oxidized to a depth of less than 0·003 in. (0·075 mm.). Details are given below.

Ni %	Cr %	Ge %	Li %
30	40	30	—
38·1	28·6	28·6	4·7
28·6	19·1	47·6	4·7
19·1	19·1	57·1	4·7

The test to determine the ductility of a brazed joint in sheet material was evolved on the premise that the load necessary to cause cracking in the fillet of the braze may be correlated with the ductility of the brazing alloy. The results exhibit a wide range of scatter, but statistical analysis of the data has shown the test to be capable of differentiating between different brazing alloys. Preparation of the 'Inconel'/stainless-steel specimen is illustrated and details are given of the procedure used.

Welding of Dissimilar Steels and Alloys

M. C. T. BYSTRAM: 'Some Guidance on Welding Dissimilar Alloy Steels.'

Brit. Welding Jnl., 1958, vol. 5, Oct., pp. 475–80.

Recent trends in the fabrication of industrial plant have led to increased use of welding for joining materials of different compositions. In many cases such combinations are preferred as simplifying design and effecting economy. Welding of clad steels is a specialized example illustrating this trend.

Although some dissimilar-metal joints present no major difficulties, certain combinations give rise to trouble, due to thermal-fatigue or stress-corrosion effects. In this paper a review is made of the problems which may arise in joining various types of low- or high-alloy steels and high-nickel alloys, the properties required in dissimilar-metal joints, and methods of securing the best results.

The make-up of any typical dissimilar-metal weld is considered in relation to its component parts, and mention is made of additional complications which may be introduced by the insertion of 'buffer layers' of still different compositions, which may be introduced between the two parent materials. Consideration is given to the nature and inherent characteristics of the base metals, the heat-affected zone, and conditions at the junction of the base metals and the weld metal, as affecting precautions required in welding and the properties of the joints produced. Special recommendations are made also with regard to deposition of the first (stress-carrying) runs, and on selection of composition of the central (undiluted) runs of multi-pass welds.

The foregoing general discussion on possible sources of weakness in mixed welds is followed by sections dealing with specific types of dissimilar-metal joints:

(1) *Welding Dissimilar Low-Alloy Ferritic Steels*, for use in ambient, sub-zero or elevated-temperature conditions, and calling for varying degrees of tensile strength and impact-resistance, associated with certain other specified metallurgical and physical characteristics.

(2) *Welding Mild and Low-Alloy Steels to Various Grades of Stainless Steel.*

(3) *Joining High-Nickel Alloys to Other Materials.*

In each section a table summarizes welding materials and procedure which have been found suitable for joining the dissimilar metal discussed.

Electrodes and Welding Wire for Welding Dissimilar Metals

INTERNATIONAL NICKEL CO., INC.: 'Joints made with 'Inco-Weld A' and 'Inco-Weld A' Wire.'

Publn. issued 1958; 10 pp.

This report is a compilation of information derived from laboratory and field experience obtained with a welding electrode and filler wire specifically developed for welding dissimilar materials.

The difficulties inherent in making such joints are reviewed, and the properties required in filler metals for making dissimilar joints are listed: see below.

(1) High tolerance for dilution by elements such as iron, nickel, chromium and copper, without forming brittle or crack-sensitive alloys.

(2) Moderate coefficient of thermal expansion.

(3) Ability to withstand high service temperatures over long periods, without harmful effects such as formation of sigma phase and migration of carbon.

(4) Good corrosion- and oxidation-resistance.

(5) High strength and ductility, at both high and low temperatures.

With these requirements in mind, The International Nickel Company has carried out an extensive programme planned to develop a coated electrode and a bare filler wire which would be satisfactory for joining combinations of high-nickel alloys, stainless steels and low-alloy steels. The work has resulted in 'Inco-Weld A' electrode and 'Inco-Weld A' wire, the properties and applications of which are discussed in this publication.

The nominal compositions of the two materials are shown in the table below.

Among the data tabulated in the publication are mechanical properties of all-weld-metal deposits, normal and elevated-temperature properties of joints made with the respective materials, and impact data showing the suitability of the new welding media for joining steels to be used at sub-zero temperatures. A list of typical joints made with the 'Inco-Weld A' electrode or wire includes over 40 dissimilar-metal welds involving ferrous/ferrous and ferrous/non-ferrous combinations. The new materials are useful also for similar-metal joints of many types.

Welding of Nickel and High-Nickel Alloys

J. HINDE: 'Welding of Nickel and High-Nickel Alloys in the Chemical Industry.'

Brit. Welding Jnl., 1958, vol. 5, July, pp. 311–18.

The paper reviews the metallurgical factors involved in welding nickel and high-nickel alloys, and outlines the welding procedures which experience has proved suitable for fabricating chemical plant from such materials. Discussion is limited to nickel and alloys of nickel-copper, nickel-chromium-iron, nickel-molybdenum, nickel-molybdenum-chromium and nickel-molybdenum-chromium-iron base. These materials, (compositions and typical properties of which are tabulated), are briefly introduced by notes on their corrosion-resistant characteristics and the applications which they have found in the chemical industry.

Nickel and the alloys discussed may be welded by all the conventional techniques, but those generally used for chemical plant are the oxy-acetylene, metal-arc and inert-gas metal-arc processes. Although no official specifications are available with respect to filler wires or electrodes for the nickel-chromium-molybdenum-iron alloys, those for nickel and the other alloys are covered by A.W.S./A.S.T.M. specifications. Designations and compositions of electrodes and filler wire for these materials are summarized, in tabular form, for each of the three welding techniques. Discussion of the metallurgical factors involved in welding nickel and the alloys considered relates mainly to selection of the appropriate electrode. Emphasis is placed throughout on conditions which will ensure maintenance of corrosion-resistance in the weld and the heat-affected zone. A subsequent section is concerned with actual welding practice. Joint preparation is illustrated, and a brief résumé is given of procedures recommended for oxy-acetylene, argon-arc and metal-arc welding. Attention is also directed to features of weld quality which are specially applicable to fabrication of chemical plant: uniformity of penetration, surface finish, careful handling, and removal of flux or slag.

In the final section of the paper welding sequences suitable for use in cladding steels with nickel, nickel-copper and nickel-chromium-iron alloys are outlined, and reference is made to methods employed for lining steel vessels, and for overlaying.

Electrodes for Welding Niobium-Stabilized Austenitic Steel for Steam Plant

R. D. WYLIE: 'Co-operative Investigation of a New Welding Electrode for Stainless Steel.'

Welding Jnl., 1958, vol. 37, Sept., pp. 426s–32s.

In the literature of the past few years there have been many references to difficulties experienced in welding of stainless steel of the A.I.S.I. Type 347*, which is extensively used for piping in steam-power generating stations, and has been adopted for various purposes in nuclear power plants. The background to the problem, its practical significance, and attempts which have been made to solve it, are concisely reviewed as an introduction to this report. One line of approach has been to determine the basic cause of the weld cracking liable to occur with this type of steel and thus, if possible, improve the steel by modification of composition. The second line has centred on improving the hot ductility of the weld and weld-affected zone, so increasing the ability

*Carbon 0·08 max., chromium 17-19, nickel 9-12, niobium 10 × C min., per cent.

	C %	Mn %	Fe %	S %	Si %	Cu %	Al %	Ti %	Cr %	Nb %	Mo %	Ni+Co %
'Inco-Weld A' Electrode (all-weld-metal deposits)	0·15 max.	1·0-3·5	6·0-12·0	0·02 max.	0·75 max.	0·5 max.	—	—	13·0-17·0	1·0-3·0	0·5-2·0	balance
'Inco-Weld A' Wire	0·07 max.	2·0-2·75	8·0 max.	0·01 max.	0·15 max.	0·20 max.	0·10 max.	2·75-3·35	15·0-17·0	—	—	balance

of the joint to relax at high temperature without cracking.

Development, some five years ago, of a new welding electrode containing chromium 16, nickel 8, molybdenum 2, per cent. aroused considerable interest because it was found by several investigators (and in service conditions) that joints made with an electrode of that composition gave more satisfactory properties. In the interim, various research and industrial organizations have carried out extensive tests on the new electrode, and have recently supplied their reports for summary and correlation. This information forms the basis of the present paper.

The subject is discussed in sections covering, respectively, tensile tests, impact tests, high-temperature properties, corrosion-resistance, physical properties, and microstructures. and some particulars are given of simulated-service tests and plant-service experience. The information, presented in graphical and tabular form, is essentially in the nature of reference data, and is necessarily too condensed to permit of detailed extraction. The following notes serve, however, to give the trend of the conclusions reached.

Tensile Tests

Extensive tests on 16-8-2 weld metal, in the as-welded condition and after various post-weld thermal treatments (including long-time ageing at elevated temperatures), indicate that post-weld solution treatment lowers the tensile and yield strengths of the deposited metal, with simultaneous increase in elongation and reduction of area. Ageing at elevated temperatures has a similar effect on the as-welded tensile properties, without increasing ductility. There was slight increase in yield strength of solution-heat-treated weld metal after ageing, but ductility remained in the same range as for unaged specimens.

Impact Tests

After ageing at the most severe temperatures used (1200° and 1350°F.; 650° and 730°C.) the weld metal in both the as-welded and solution-treated conditions maintains an impact strength of more than 20 ft.-lb. (2·76 kgm.). Indications are that at 1500°F. (815°C.) the sigma phase which forms does not have so pronounced an effect on impact properties as at lower temperatures. Comparison of the impact strength of the 16-8-2 welded deposits with that of other commercial chromium-nickel stainless-steel deposits has shown that the 16-8-2 weld retains a higher level of impact strength than other chromium-nickel stainless-steel deposits when aged without post-weld heat-treatment.

High-Temperature Properties

In these tests comparison was made of the properties of niobium-stabilized or molybdenum-containing wrought steels, or with commercially available weld-deposit metal of those types. Evaluation was by the hot-ductility tensile test developed by Nippes. Stress-rupture properties were studied over the range 1050°-1500°F. (565°-815°C.): the results show that in the range 1050°-1350°F. (565°-730°C.) the weld deposit is stronger than the wrought parent material, but that at 1500°F. (815°C.) the reverse is true. At all test temperatures the solution-treated weld deposit had lower strength than the as-welded deposit. Creep strength of the as-welded deposits was found to be considerably higher than that of the base material. The relaxation-test data indicate that the 16-8-2 composition tends to flow or relax at lower stress levels than those causing relaxation in 19-9 chromium-nickel-niobium steel, and that solution treatment improves the ability of the weld deposits to relax. (It has been postulated that one of the most important considerations in the successful performance of an austenitic weld is the capability of the residual stresses introduced in welding to relax at a rate sufficiently rapid to avoid stress-rupture failure adjacent to a weld.)

Corrosion-Resistance

Conventional boiling HNO_3 (Huey) and acidified $CuSO_4$ tests, and air-oxidation tests, showed that the 16-8-2 weld deposit reacts in a manner very similar to that observed in molybdenum-containing wrought steel. After ageing at temperatures of 1050°-1350°F. (565°-730°C.) the material is subject to rapid intergranular corrosion under severe service conditions, but after prolonged periods at temperature stabilization occurs, through diffusion, with the result that material which has been aged at 1000 hours at 1350°F. (730°C.) resists intergranular attack. At temperatures up to 1350°F. (730°C.) the oxidation-resistance of the 16-8-2 weld deposit is similar to that of wrought molybdenum-containing stainless steel, but at higher temperatures more rapid oxidation occurs in the weld deposit, probably due to its lower chromium content.

Physical Properties

A curve relating magnetic permeability to ageing time shows that with prolongation of ageing magnetic permeability increases to a maximum and subsequently falls to near the original level. This curve parallels the effect of carbide precipitation on intergranular corrosion-resistance, thus confirming the re-distribution of chromium which occurs. The thermal-expansion characteristics of the 16-8-2 weld deposit are similar to those of conventional austenitic wrought steels.

Microstructure

The composition of the 16-8-2 weld deposit is balanced to provide a minimum of ferrite: in most cases the ferrite content is in the range 0-5 per cent. On ageing, the weld deposit reacts in a manner similar to that observed in molybdenum-containing 18-8 steel, in that a certain amount of sigma phase and carbide is precipitated. The quantity of sigma phase produced is, however, less, due to the lower chromium and molybdenum contents.

Simulated-Service Tests and Service Experience

In order to establish evidence on the new electrode composition, simulated-service tests have been run and some are still in progress. Experience to date has been generally satisfactory, although some

cracking has developed. It is concluded that in welding niobium-stabilized steel it is not possible completely to overcome cracking by changing to the 16-8-2 electrode. Some change in composition of the wrought material for piping will probably be required, but it is clear that the 16-8-2 electrode makes an important contribution towards solution of the problem.

Binders bearing the date 195- and suitable for 1958 and 1959 Nickel Bulletins are now available.
We shall be pleased to send one free of charge.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks.*

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SUBJECT INDEX TO VOLUME 31

The Index comprises a consolidated list of titles of abstracts, compiled from the sectional contents lists of the individual issues.

Within the Sections normally used (**GENERAL, NICKEL, ELECTRODEPOSITION,** etc.), the items are broadly classified in appropriate sub-sections, within which the items are, in most cases, entered in the order of their appearance in the Bulletin. The only exceptions to such order are (1) the grouping of the page numbers of serial items, and of items of very closely related subject matter, and (2) the alphabetical order used in the **GENERAL** Section.

The Leading Article referred to below, although listed separately, is shown also in the appropriate sections, where it is differentiated from the abstracts by an asterisk at the beginning of the entry.

A list of **Trade-Named Materials and Processes** referred to in this volume of the Bulletin appears on p. 369.

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